Study on Phases Development and Mechanical Properties in a Fe-Ni-Al Carbide Free Bainitic Steel Based on Lateritic Steel After Warm Rolling

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Abstract. The abundant reserve of lateritic ores in Indonesia is currently processed and studied to fulfil the national steel demand in several sectors, one of which is for railway application. The development of lateritic steel (Fe-Ni) as carbide free bainitic steel is carried out by adding Al to the Fe-Ni alloy. In this research, the effect of warm rolling and Al addition to the formation of carbide, phases development and mechanical properties was studied. The warm-rolled thermomechanical process (TMCP) was carried out by heating the material at 945°C for 20 minutes followed by second heating to 400°C, 450°C and 500°C with holding time for 30 minutes. The materials then warm rolled with 50% and 70% reductions using 20 tons capacity roller machine and then air cooled outside the furnace chamber. The microstructure of the as-rolled materials was characterized using optical microscope (OM) and scanning electron microscope (SEM), while the phases, the chemical distribution and the possibility of carbide formation were examine using X-Ray Diffraction (XRD) pattern and energy dispersive X-Ray spectroscopy (EDS). The mechanical properties of the material were observed using macro Rockwell hardness test. It was revealed that the addition of Al altered the phases of Fe-Ni lateritic steel significantly. Furthermore, Al addition gives positive effects to the Fe-Ni lateritic steel by increasing hardness. The reductions applied during warm rolling were observed to have effect on the growth of the grains in the Fe-Ni-Al lateritic steel.

1. Introduction
Indonesia is one of countries that has the largest laterite iron ore reserves in the world spread in South Kalimantan, South Sulawesi, Southeast Sulawesi, North Maluku and West Papua. The development of laterite (nickel) steel ore can be used as an alternative to reduce the dependence on steel raw materials, especially raw materials for automotive in Indonesia. One development of Fe-Ni based laterite steel is by adding alloy and microstructural engineering with the thermomechanical control process (TMCP) process. The purpose of this development is to get a new type of steel, which is known as carbide free bainitic steel, in which it is an interesting candidate for automotive and railway industries application[1,2], which exhibits good combination of strength, toughness and ductility, has been designed and manufactured [3-6].

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The previous research reported that the microstructure of carbide free bainitic steel consists of fine plates of carbide-free bainitic ferrite and thin film of carbon which is enriched by retained austenite[6]. Fine-scale microstructure is often obtained by the bainite transformation with the diffusion less growth of tiny bainitic ferrite platelets that is mostly gotten at low temperatures, and it is primarily achieved in steels with a higher carbon content, in which does not only decrease the bainite transformation temperature, but also stabilizes retained austenite after bainitic transformation [4-7].

Adding the aluminum in the steel can accelerate the low temperature of bainitic transformation, especially the formation of bainitic ferrite [4,8]. C.W. Shao et al [9] studied that the addition of Al does not only increase the stability of α-ferrite but also expands the δ-ferrite phase region. Aluminum has a similar function to suppress the precipitation of cementite to silicon. It also has been observed that adding aluminum to TRIP Steels tends to refine the bainitic lath structure [8,10]. The presence silicon in alloy will affect the microstructure. It was found that silicon can only impede cementite (Fe₃C) formation lower bainite but does not fully suppress it [11]. Producing low carbon bainitic steels in the Fe-Mn-Ni-C was also investigated and found that the platelets of bainitic ferrite were extremely coalesced and the coarsening of microstructure occurred. Thus, it eliminates the beneficial effect of fine scale bainitic microstructure.

Warm rolling has a potential process to get such beneficial microstructure as the structure of the ultrafine-grained ferrite matrix to offer considerable costs savings due to lower reheating temperatures, reduce the roll wear and loss of scale, and allow accurate control of product dimensions [12]. W. Hui et al [11] studied the microstructural evolution and mechanical properties of a 3% Al containing medium Mn steel during warm rolling after intercritical annealing at temperature 750°C and found that the ferrite and retained austenite lath changed gradually to follow rolling direction and refined by increasing the rolling thickness reduction. Rolling effect indicates that the formation mechanism of the lamellar structure is a kind of grain subdivision, the deformation induces grain boundaries subdivide the initial grains to ultrafine regions [13].

Current study is aimed to explore of possibility of formation of the carbide free bainitic microstructure in a lateritic steel alloyed with 2,23 wt. % Al and examine the effect of austempering temperature and warm rolling by reducing the different thickness after austempering on the microstructure and hardness of this steel.

2. Experimental Details
The chemical composition of the Fe-Ni lateritic steel alloyed with Al is given in Table 1. The dimension of the sample used in this study is 75 x 20 x 10 mm were cut to austempering treatment and warm rolled. Before the thermochemical treatments were conducted, the bainitic start temperature (Bs) was calculated to be 579°C for Al-added steel, while the martensitic start temperature (Ms) 418°C. The calculation method occupied was the MUCG83 program developed by Bhadeshia at Cambridge University. During the thermomechanical treatment, the samples were heated to 950°C for 20 mins to obtain full austenite structure, followed by furnace cooling to reach bainitic transformation temperature and hold for 30 mins. Three different bainitic transformation temperatures were used, which are 400°C, 450°C and 500°C. At each of these temperatures, the sample was subjected to warm rolling with 50% and 70% reductions using 20 tons capacity roller machine. Then, the samples were air cooled to room temperature outside the furnace chamber. The schematic image of the thermomechanical schedules is shown in figure 1. The orientation corresponding to warm rolled product are shown in figure 2, in which ND stands for normal direction, TD stands for transverse direction and RD stands for rolling direction.

| C   | Si  | Al  | P   | S   | Cr  | Ni  | Mo  | Mn  | Fe  |
|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| 0.30| 1.38| 2.23| 0.0005| 0.0003| 0.28| 3.03| 0.20| −0.4| Bal |

Table 1. Chemical composition of the experimental steel (in wt.%):
Figure 1. Schematic illustration of the thermomechanical schedules applied to studied samples.

Figure 2. Corresponding directions of the warm rolled product.

3. Result and Discussion

3.1 Microstructure analysis

3.1.1. Microstructure

The longitudinal sections (along rolling reduction) of the samples were mounted and polished using the standard metallurgical procedure and etched in 4% picral, and their microstructure were observed using an optical microstructure (OM) and a scanning electron microscope (SEM, JEOL 6390). The heat-treated samples were also characterized using X-Ray Diffraction (XRD Rigaku Miniplex 600) pattern diffraction instrument with Cu Kα radiation analyzing between 0° and 125° (2θ), and energy dispersive X-Ray spectroscopy (EDS) to examine the chemical distribution and the possibility of carbide formation. Macro hardness test was carried out on ND of warm rolled samples. Macro hardness measurements were conducted using a load of 150 kgf on a Rockwell hardness testing machine with diamond indenter and the procedural based on ASTM E18. At least six reading were taken for each heat treatment condition and their average is reported here as the hardness value.

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elongated martensite or retained austenite (M/RA) particles, in which generally surrounded by bainite grains. Isothermal treatment without holding time at 400°C followed warm rolling 50% (figure 3b) shows the microstructure consisted of mainly bainite and ferrite. Holding time is affected by the bainite transformation of microstructure, when the period of holding is short, the amount of bainite formed at the end of the holding period is small. As can be seen, the size of grain is thin and it is discontinuous surrounded by acicular ferrite grains. When the isothermal treatment has 30 min holding time the mainly result of microstructure consists of bainite ferrite and ferrite with less or no martensite (figure 3c-3g). By increasing the temperature, the amount of bainite increases, while the amount of ferrite decreases. The bainite ferrite and ferrite are arranged alternatively along the rolling reduction.

Figure 3. SEM micrographs of the samples, (a) air cooling condition after austenization 945°C; (b) WR 50% at 400°C without holding time; WR 50% after holding time for 30 mins at (c) 400°C, (d) 450°C, (e) 500°C; WR 70% after holding time for 30 mins at (f) 400°C, (g) 450°C, (h) 500°C.

The effect of rolling different rolling reduction on warm rolling microstructure are illustrated in figure 3c-3g. The microstructure will develop lamellar structure with the longitudinal axis along rolling reduction. The bainite ferrite and ferrite phases gradually changed their longitudinal axis to the rolling reduction by increasing the amount of deformation. The microstructure (figure 3c-3g) shows that ferrite
changed from figure 3a to be elongated ferrite along the rolling reduction. It is obvious that ferrite phase was significantly elongated and refined with highly rolling reduction at WR 70%. In fact, the microstructure becomes finer with increasing of warm rolling reduction (Figure 3c-3g).

3.1.2. Energy Dispersive X-ray spectroscopy (EDS) Analysis
The microstructure of the SEM micrograph to EDS characterization is shown in figure 4. There are dark particles that indicated the existence of carbide and bainite and ferrite that is masked in grey. The result of EDS characterization shows that dark particle consists of aluminum compound as much as 59% in figure 4a and 61% in figure 4b. The amount of aluminum slightly increases following the rising of warm rolling reduction. The raising of warm temperature rolling will increase the content of aluminum compound up to 92% in dark particle. This is caused by carbide is not being transformed at high temperature and producing the granular microstructure. The grey microstructure consists of mainly Fe compound which consists of ferrite and bainite phase. The amount of carbon contained in the ferrite phase is less than in the bainite phase and increase following the temperature.

Furthermore, the result of EDS analysis depicts that the effect of increasing warm rolling reduction does not only increase aluminum content but also decreases nickel contained in ferrite phase. In bainite phase, aluminum content decreases while nickel content increases with increasing warm rolling reduction. Increasing temperature of warm rolling will decrease the fluctuated aluminum and nickel contained in ferrite and bainite. However, the highest aluminum content is found at the lowest temperature warm rolling.

![Figure 4. SEM micrographs to EDS characterization of the samples, (a) WR 50% for 30 min at 400°C, (b) WR 70% for 30 min at 400°C](image)

3.1.3. X-Ray Diffraction (XRD) Analysis
Typical XRD patterns is shown in figure 5, in which all peaks only consist of α phase. The integrated intensity ratio of α (110) peaks is much higher than other peaks in the samples. The intensity of α (110) increases when warm rolling temperature decreases. The α (310) peak slightly increases when warm rolling temperature decreases. Furthermore α (200), α (211) and α (220) peak increase fluctuate and tends to be stable without being affected by temperature.

The effect of warm rolling with 50% and 70% reduction on XRD pattern shows the integrated intensity ratios α (110) decreases when the warm rolling reduction increases. The α (200) and α (211) peak fluctuated as the effect of increasing warm rolling reduction. From typical XRD pattern, it can be concluded that there is no carbide form and further confirmation that only bainite and ferrite phases exist in the samples.
Figure 5. XRD patterns of lateritic steel alloyed with Al sample (a) WR 50% at 400°C, 450°C and 500°C, (b) WR 70% at 400°C, 450°C and 500°C.

3.2 Mechanical Properties
3.2.1. Hardness Measurements
The variety of macro hardness of as cast and warm rolled steels with different reduction are shown in figure 9. Sample with austenization treatment at 945°C followed air cooling and no warm rolling has lower hardness (3.37 HRC) than other samples. This confirms that microstructure of sample No WR consists of M/RA, ferrite and less bainite ferrite. Sample with isothermal treatment without holding time followed WR 50% has 17.23 HRC. The hardness of sample that is no holding WR 50% increases because the warm rolling treatment and the amount of bainite ferrite increase while the amount M/RA decreases.

Figure 9 also shows the value of hardness increase significantly with warm rolling treatment. The hardness of 70% warm rolling reduction is higher than hardness of 50% warm rolling reduction. The deformation effect increases dislocation density and the material hardening by inhibiting the movements of dislocation. From the results of optic and SEM photographs, shows that warm rolling reduction changes in grain shape from equiaxed to elongated and refined. These internal changes have led to increase the hardness and strength. With the increasing of deformation, the stored energy increases and changes the driving force for recrystallization and form many new nucleation. Furthermore, figure 9 shows that the hardness increases as the transformation temperature reduce. It confirms that hardness increases when the less refinement of bainitic ferrite is detected on microstructure.
4. Conclusions
The heat treatment, warm rolling process, microstructure and mechanical property of lateritic steel alloyed with Al was investigated. This study found that the microstructure of bainite ferrite and ferrite was obtained, and the precipitation of carbide was reduced. This indicates the addition of aluminum effectively suppresses carbide formation during bainite transformation. The results of XRD show that no carbide form and further confirmation that only bainite and ferrite phases are present in the sample. Effect of warm rolling, the microstructure of lateritic steel changes from equiaxed to lamellar structure bainite ferrite and elongated ferrite with the longitudinal axis along rolling reduction. It is obvious that the microstructure becomes finer with increasing warm rolling reduction. The hardness was found to increase with increasing warm rolling reduction and decreasing temperature warm rolling. The maximum hardness value is 33.62 HRC obtained isothermal treatment at 400°C for 30 min followed by 70% warm rolling reduction.

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