The effect of hot rolling on hardness properties and microstructure of laterite steel

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Abstract. Nickel containing pig iron (NCPI) is one of important materials for stainless steel and other iron-nickel alloys production. The natural source of NCPI in Indonesia is laterite ore. Large deposit of laterite ore has been found in South East Sulawesi. High grade laterite ore (saprolitic type of laterite ore) in this region has been used for ferronickel making, whereas low grade laterite ore (limonitic type of laterite ore) has not been processed, due to its too low nickel content. However, laterite steel has not been widely reviewed as potential candidate for top national product comprehensively. Therefore, this study is aimed to know several aspects of its intrinsic properties by using hot roll process. A variety of thickness reduction on steel is used by rolling to know its mechanical properties especially hardness, followed by microstructure observation, and then compared with its as-cast. The result has shown that the highest hardness (282 HRB) obtained in 31-35% reduction which is increased by 47.7% of as cast steel. Optical microscope images showed that ferrite phase and grain refinement which is caused an increasing on hardness. This study is expected as a reference for further research. The chemical composition of steel Laterite as more likely as HSLA steels with certain specialized alloys to obtain specific mechanical properties. Meanwhile the hardness of Laterite steel is possible for some products similar to AISI416 standard.

1. Introduction
Laterite iron is a type of residual sediment produced from the weathering process of rocks which involves decomposition, re-deposition, and chemical collection. Laterite ore is generally found in the peak areas of hills with a <10% slope. This slope is a main factor in it's chemical weathering process, which has a greater role than the mechanical process. Meanwhile the structure and characteristics of the soil is affected by mineral solubility and groundwater flow conditions [1,2,4,6].

The properties of laterite deposit is that it has a texture that is clearly blatant, the coating is compact, the mineral composition of iron varies, the level of Fe content ranges between 40.00 and 60.00%, contains lower levels of Ni and Cr than laterite, containing an average of 0.41% Ni and 2.10% Cr$_2$O$_3$, especially from laterite ore, may contain bog iron ore, with a high levels of sulfur and manganese, while those originating from hot springs can contain relatively higher levels of sulfur, and the Al levels are lower than lateritic types, being at around 7.00%, [3,6,9,12].

Numerous laterite ores are found in several places in Indonesia, such as Sebuku, Mt. Kukusan, Geronggang (South Kalimantan), Pomala (Southeast Sulawesi), and Halmahera. It is estimated that the amount of laterite ore deposits reaches up to 950,000,000 tons with Fe levels at 39.8 - 55.2%. The
characteristics of laterite ore are low levels of iron and contains impurities such as nickel, chromium, cobalt, manganese and high levels of water content. With such characteristics, laterite ore has yet to be utilized in the iron-steel industry. In general, the steel industry requires 60-69% levels of iron content. At the moment there is no effective and economical technology to increase the levels of Fe in lateritic ore, that can meet the requirements for it to be used as raw material in the steel industry. [2,5,8,10]

There are several technologies that have been implemented and are quite successful in increasing the levels in hematite and magnetite types of iron ore. The grade increase method applicable is called the magnetizing roasting method which in general uses natural gas as a reducing agent. Natural gas is converted into CO and CO2 in a reformer and is injected into a reduction furnace and flotation method for both foam flotation and reverse flotation. In the flotation method the iron mineral froth is floated as a concentrate while in reverse flotation, the dirty minerals in the form of silicates are floated as concentrate [3,6,8]

From the chemical analysis it is found that the Fe contained in laterite ore is 41.88%, 0.499% Ni, 0.04% Co, 1.26% Cr, 9.46% Al2O3, 18.47% SiO2. Beneficiation by the magnetizing roasting process produces Fe in magnetite separator concentrate at 66%. Fe acquisition reached 65.66%. The new silica content can be reduced from 18.47% to 10.65% and the Al2O3 content of 9.465 decreases to 6.57%. Meanwhile the composition of the pulp is: 34.67% Fe, 18.70 SiO2, 16.21% Al2O3, 1.27% Cr, 0.013% Co and 0.17% Ni. At optimum conditions the flotation experiment produced concentrates containing 57.90% Fe, 0.3% Ni, 6.30% SiO2 and 2.06% Al2O3, while in the pulp there is 20.96% Fe and 0.7% Ni. The optimum conditions in the reverse flotation experiment resulted in concentrations of 71.74% Fe.

The problem of the impurity content in the form of SiO2, Al2O3, Cr2O, Ni and Co means that it still cannot meet specifications for it to be used as raw materials. The low Fe gain at 58.76% is an obstacle in applying beneficiation technology both flotation and magnetizing roasting. The sponge iron produced has a total Fe content of 75.9% and the pigiron at 95% Fe making them optimum enough which shows that the reduction process can take place well. [5,8,9,11,12]

1.1. Hot Roll
Rolling is the process of deformation with certain condition of temperature and pressure that can be affected the grain size reduction. There are two type of rolling based on work temperature, hot and cold roll. Cold roll is rolling process by using temperature under conditions of recrystallization which is to increase the strength by utilize the effect of strain hardening[4]. While hot roll is working temperature is above of recrystallization temperature that to obtain fine the grains. In addition, hot roll is also to homogenize the grain structure for casting [5,9,12,15].
2. Research Method
The laterite steel in this research was obtained from NPI (nickel pig iron) where laterite steel was added to other chemical elements in the form of carbon, manganese, chrome, nickel where the composition or mixture of these chemicals was mixed during the melted laterite steel casting process, the mixture included: Carbon: 0.1412; Nickel: 2.5583; Chrome: 1.3561; Manganese: 0.4544. From the data/mixture of carbon above, laterite steel can be classified into low carbon steel.

![Figure 1. Recrystallization scheme [6].](image)

![Figure 2. Research Method Flowchart](image)
3. Result and Discussion

| No | Sample  | Reduction (%) | Testing 1 (HV) | Testing 2 (HV) | Testing 3 (HV) | Average (HV) |
|----|---------|---------------|---------------|---------------|---------------|--------------|
| 1  | Sample 1 | 0  | 299 | 204 | 286 | 263 |
| 2  | Sample 2 | 5  | 286 | 208 | 345 | 279 |
| 3  | Sample 3 | 10 | 302.5 | 204 | 341 | 282 |
| 4  | Sample 4 | 15 | 247 | 206 | 341 | 264 |
| 5  | Sample 5 | 20 | 257 | 193,5 | 252 | 234 |
| 6  | Sample 6 | 25 | 246 | 192 | 349 | 262 |
| 7  | Sample 7 | 30 | 236 | 192 | 313 | 247 |
| 8  | Sample 8 | 35 | 208 | 195 | 299 | 234 |
| 9  | Sample 9 | 40 | 206 | 186,5 | 293 | 228 |
| 10 | Sample 10 | 45 | 202 | 193,5 | 249 | 214 |
| 11 | Sample 11 | 50 | 232 | 195 | 371 | 266 |

The graph above portrays the comparison of the hardness test between reductions. The highest hardness value occurred in sample 3 with an average value of 282 with a reduction of 10% and the lowest average value in the Vickers hardness test occurred in sample 10 with an average value of 214 with a reduction of 45%.
3.1. Microhardness test results

Tabel 2. Microhardness tests on laterite steel

| No | Sample  | Reduction | 1 (HV) | 2 (HV) | 3 (HV) | Average (HV) |
|----|---------|-----------|--------|--------|--------|--------------|
| 1  | Sample 1| 0         | 311,6  | 296,4  | 307,1  | 305          |
| 2  | Sample 2| 5         | 295,3  | 280,3  | 294,3  | 290          |
| 3  | Sample 3| 10        | 262,9  | 270,9  | 339,3  | 291          |
| 4  | Sample 4| 15        | 272,2  | 304,9  | 283,2  | 287          |
| 5  | Sample 5| 20        | 285,2  | 251    | 289,2  | 275          |
| 6  | Sample 6| 25        | 264,3  | 252,3  | 302,1  | 273          |
| 7  | Sample 7| 30        | 245,5  | 250,3  | 296,5  | 264          |
| 8  | Sample 8| 35        | 252,5  | 238,5  | 294,3  | 262          |
| 9  | Sample 9| 40        | 251,3  | 237,5  | 290,6  | 260          |
| 10 | Sample 10| 45       | 247,3  | 236,5  | 246,3  | 243          |
| 11 | Sample 11| 50       | 247,5  | 238,3  | 349,5  | 278          |

Figure 4. Microhardness hardness test on laterite steel.
From figure 4 it is clearly observed that the greater the reduction given to the sample, the lower the resulting hardness. This is influenced by grain boundaries that are getting farther or more visible which causes the material to get brittle but hard (not ductile). This is influenced by the imposition of a larger force given to the sample hence the results. These errors include surface smoothness, porosity, type of treatment, and differences in alloying elements \cite{3,12,13,14}.

The influence of Ni based on equilibrium diagram of Fe-Ni shown in Fig.5. Nickel have been soluble in ferrite form the FCC unit cells (austenit stabilizer) and decrease the eutectoid temperature \cite{11}. It also triggers the recrystallization which produce the grain refinement caused by hot deformation \cite{12}.

![Figure 5. Phase diagram of Fe-Ni \cite{11}.

3.2. Metallography Observation Results

Metallography is a discipline that studies observation or examination methods with the aim of determining or studying a metal’s, alloy’s, or material alloys’s relationship between it’s structures with nature and character and the treatment it went through. Metallography also includes examination or observation of Crystal structures using techniques such as x-ray diffraction. However, the most commonly used tool in metallographic observation is light microscopy or optical microscopy with magnifications ranging from 50 times to 1000 times and the ability to separate or resolute microstructure around 0.2 microns or greater \cite{2,5,9,10}.

![Figure 6. Photo of sample 1 before reduction](image_url)
Figure 6 (a) shows laterite steel without being reduced or at 0% reduction rate. Figure (a) is taken macroscopically, to examine the microstructure in which it is not possible to see any transformation in the microstructure. Figure 6 (b) with 100 times magnification, the microstructure is visible but not as detail for it to be analyzed properly. Figure 6(c) shows 500 times the magnification in which the microstructure of the laterite steel is clearly visible with 0% reduction. Here we can see grain boundaries and carbon atoms being reduced by 0%. The grain boundaries that are visible are still not moving or deforming and the carbon structure seen has not occured deformation. The ferrite structure still dominates the microstructure of laterite steel which is shaped like spheres (black). [3,12,15]

![Figure 6](image)

**Figure 7.** Photo of sample 2 with 5% reduction

Figure 7(a) shows laterite steel with 5% reduction. Figure (a) is taken macroscopically, to examine the microstructure in which it is not viable to see any change that has occured in the microstructure. Figure 7(b) shows 100 times magnification in which the microstructure is not visible hence it cannot be analyzed, while in Figure 7(c) with 500 times magnification, the microstructure is visible and has begun to change shape from ferrite structured to partly pearlite structured[4,8]

![Figure 7](image)

**Figure 8.** Photo of sample 3 with 10% reduction

Figure 8 (a) displays laterite steel which has been reduced with 10% reduction. Figure (a) is taken macroscopically, in which visibility wise, no change has occured in the microstructure. Figure 8(b) shows 100 times magnification in which the microstructure is not visible hence unobservable, while in Figure 8(c) with 500 times magnification, the microstructure is visible and the latterite structure has formed into pearlite[3,14]
Figure 9. Photo of sample 4 with 15% reduction

Figure 9(a) shows laterite steel with 15% reduction. Figure (a) is taken macroscopically, to examine the microstructure in which it is not possible to see any transformation in the microstructure. Figure 9(b) shows 100 times magnification in which the microstructure is not visible hence it cannot be analyzed, while in Figure 9(c) with 500 times magnification, the microstructure is visible and shows that ferrite and pearlite still dominate the microstructure in a laterite steel with 15% reduction.

Figure 10. Photo of sample 5 with 20% reduction
Figure 10(a) shows laterite steel with 20% reduction. Figure (a) is taken macroscopically to inspect the microstructure, in which no transformation can be seen in the microstructure. Figure 10(b) shows 100 times magnification in which the microstructure is not visible hence change in micro structure is not perceptible, while in Figure 10(c) with 500 times magnification, the image shows that the microstructure in still dominated by ferrite and pearlite.

Figure 11. Photo of sample 6 with 25% reduction

Figure 11 (a) shows laterite steel with 25% reduction. Figure (a) is taken macroscopically to inspect the microstructure, in which it is unnoticeable that transformation has occurred in the microstructure. Figure 11(b) shows 100 times magnification in which the microstructure isn’t visible meaning an analysis is not yet possible, while in Figure 11(c) with 500 times magnification, the image shows that within the microstructure, pearlite structures dominate over ferrite.

Figure 12. Photo of sample 7 with 30% reduction

Figure 12(a) shows laterite steel with 30% reduction. Figure (a) is taken macroscopically to observe the microstructure, in which no visible change has occurred in the microstructure. Figure 12 (b) shows 100 times magnification in which the microstructure is not visible hence change in micro structure is not perceptible, while in Figure 12(c) with 500 times magnification, the image shows that the pearlite structure is almost perfectly deformed with bainite structure.
Figure 13. Photo of sample 8 with 35% reduction

Figure 13(a) shows laterite steel with 35% reduction. Figure (a) is taken macroscopically to inspect the microstructure, in which no transformation can be seen in the microstructure. Figure 13(b) shows 100 times magnification in which the microstructure is not visible hence change in micro structure is not perceptible, while in Figure 13(c) with 500 times magnification, the image shows that the material is dominated by bainite structures. [3,12]

Figure 14. Photo of sample 9 with 40% reduction

Figure 14(a) shows laterite steel with 40% reduction. Figure (a) is taken macroscopically to inspect the microstructure, in which there is no transformation that is perceptible happening in the microstructure. Figure 14(b) shows 100 times magnification in which the microstructure is not visible hence change in micro structure is not perceptible, while in Figure 14(c) with 500 times magnification, the image shows that bainite atomic structure is almost perfectly deformed with the pearlite structure [4,14]
1. Figure 15. Photo of sample 10 with 45% reduction

Figure 15(a) shows laterite steel with 45% reduction. Figure (a) is taken macroscopically, to examine the microstructure in which it is not possible to see any transformation in the microstructure. Figure 15(b) shows 100 times magnification in which the microstructure is not visible hence change in microstructure is not perceptible, while in Figure 15(c) with 500 times magnification, the image shows that it contains pearlite structures at 45% reduction [4,15].

2. Figure 16. Photo of sample 11 with 50% reduction

Figure 16(a) shows laterite steel with 50% reduction. Figure (a) is taken macroscopically to view the microstructure, but no transformation can be seen in the microstructure. Figure 16(b) shows 100 times magnification in which the microstructure is not visible hence change in microstructure is not perceptible, while in Figure 16(c) with 500 times magnification, the image shows that it contains pearlite structures at 50% reduction [4,12].

4. Conclusion

1) In the hardness test using the Vickers method, the highest hardness value was obtained from sample 3 with a value of 282 with a reduction of 10%, meaning the percentage of steel laterite of sample 3 is greater than the sample that has not been reduced, with the difference between them being 6.74% and the lowest average value in Vickers hardness test occurred in sample 10 with an average value of 214 with a reduction of 45%. so the percentage of steel laterite of sample 10 is greater than the sample that has not been reduced, with the difference between them being 18.63%.

2) In the hardness test using the Microhardness method by using weight of 1.96N, the highest hardness value occurred in sample 1 with an average hardness value of 305 not reduced and the lowest average value in the microhardness hardness test occurred in sample 10 with an average value of
243 with 45% reduction. In the Microhardness test above, there is a percentage comparison ratio of 20.33%.

3. The observation of metalography on laterite steel samples spotted that for each reduction has a different structure where in a steel that has not been reduced up to a steel with 25% reduction possesses a microstructure of ferrite and pearlite, while bainite structure occurs in the laterite steel samples which are reduced by 30% and 35%, and also in laterite steel samples with a reduction of 40% - 50% there is a change in the microstructure forming it into pearlite.

4. With the hardness testing by the usage of 2 methods there was a significant graph, this was because at the time of the study there was no element uniformity (homogenization).

5. References

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