THE EFFECT HOLDING TIME VARIATION ON THE PROCESS OF HEAT TREATMENT AUSTEMPERING ON MECHANICAL PROPERTIES AND MICRO STRUCTURES OF LATERITE STEEL

Djoko Setyo Widodo¹, Desiran Sembiring² and Bintang Adjiantoro³

¹PhD Student Faculty of Mechanical Engineering Program of Technology
University of Malaysia and Lecturer at Universitas Krisnadwipayana, Indonesia

²PhD Student Faculty of Mechanical Engineering Program Technology
University of Malaysia

³Metallurgical Research Center LIPI, Serpong PUSPIPTK Complex

¹ djokosetyowidodo@gmail.com, ² desmuham@gmail.com,

³ bintangadjiantoro@yahoo.co.id

Abstract— The development of laterite steel is now being discussed by various research institutions. This cannot be separated from the potential of laterite-based steel into national steel. This study uses the austempering heat treatment process with variations of holding time austempering for 30, 60, and 90 minutes and using Zn (Zinc) cooling media. The characterization of laterite steel was carried out by testing hardness, impact resistance, microstructure, scanning electron microscopy-energy dispersive spectroscopy. The results showed an impact resistance value decreased 8.46 which was initially 209 J to 191.3 J. Besides that the hardness value increased 36.58% from initially 164 HV to 224 HV. Judging from its physical properties, the microstructure formed by pearlite ferrite bainite at a 90-minute holding time while the results of the fracture are included in the ductile fracture because there are dimple holes and look blurry or dark.

Keywords— Laterite steel, heat treatment austempering, hardness, impact resistance, microstructure

1. INTRODUCTION

The development of laterite steel is now a topic of discussion by various research institutions, one of which is the Indonesian Knowledge Institute (LIPI), the Metallurgical and Materials Research Center (P2MM) for its potential to become national steel. According to Barkas in Hasbi et al's 2016 study, generally, laterite nickel ore is used as an alloy in the manufacture of stainless steel, the application of superalloys and alloy metals to increase corrosion resistance. With iron content of 35-45% and nickel 0.8-1.5%, after being processed into steel, laterite nickel ore in the form of Nickel Pig Iron (NPI) will produce steel with a nickel content of 2-3% (Binudi and Adjiantoro, 2013).

The research on compartment of austempering and quenching and tempering processes for carburized automotive, explained the austempering process as a method that could possibly replace quench and tempering in the treatment of SAE 8620, 4320 and 8822 steels because this process can reduce distortion and be more economical (Andrew Clark, 2013) Najeeb Niazi (2014) also conducted a study on austempering heat treatment of

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AISI 437 in this study, the higher and longer the time the austempering can increase the hardness and impact toughness of AISI 4340 steel. The effect of the austempering process on Nodular cast iron with variation and time can produce residual austenite bainite microstructures (Furqon and Hafid, 2012). According to Zdzislaw Lawrynowicz (2016) in his study of the bainite transformation during the austempering of ductile cast iron, the results of optical microscope testing were that bainite ferrite growth could continue to a greater extent and not only pure austenite but also a mixture of ferrite bainite, austenite and carbide.

As a step to find out some of the aspects possessed by laterite steel in terms of its potential, research will be conducted one of which is through the austempering heat treatment process. With variations in holding time austempering and characterization are carried out. The austempering heat treatment process, where rapid cooling of the austenite phase to the isothermal transformation area must be as fast as possible (C-D). This is to avoid the perlite phase so that maximum toughness and tenacity can be achieved. The temperature on the isothermal transformation (D-E-G) is above the temperature of martensite starts, while the isothermal transformation takes place in two stages. The first stage (D-E) is transformed from austenite to ferrite and the target area by diffusion of C atoms into regions with stable austenite. Stage two (E-G) transforms from remaining austenite to bainite and iron carbide. Like the scheme is shown in Figure 1. In this study the temperature of martensite start can be searched by the following equation:

$$M_s (^{\circ}F) = 1000 - (650 \times \%C) - (70 \times \%Mn) - (35 \times \%Ni) - (70 \times \%Cr) - (50 \times \%Mo)$$

Fig. 1 Austempering Process Scheme (Sumber Raharjo, 2007: 45)

The appropriate heat treatment austempering process must be considered for the selection of the dye media. In the austempering process, the dyeing temperature is around 320-550°C so that a non-evaporation dyeing medium is needed at the temperature region. In the research that will be carried out the austempering cooling media used is the element Zinc (Zn) which has a melting point of 419°C and a vapor point which reaches 907°C (sciencelab.com) with the austempering process at a temperature above Ms temperature of around 250-550°C. Therefore in this study, the author discusses the effect of holding time variations on the austempering heat treatment process on the mechanical properties and microstructure of laterite steel.

2. RESEARCH METHOD

This study uses an experimental method to look for certain treatments for others in controlled conditions (Sugiyono, 2012: 72). The treatment carried out in this study is the austempering process and the results obtained in the form of Vickers hardness value, impact resistance value, microstructure formed, and the shape of the fracture that occurs.

In this study, the specimens used were laterite steel castings made at the Metallurgical Research Center and Materials of the Indonesian Institute of Sciences. The laterite steel needed in this study has a carbon composition of 0.2% wt with chemical analysis using

spectroscopy. Laterite is a result of weathering that lasts very long from ultra-alkaline rocks. One of the laterites is laterite iron ore which is the main mineral of laterite iron ore is Goethite, ($\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$) other minerals are Kwarsa (SiO_2), Hematite (Fe_2O_3), Nickel Oxide (NiO) and Chromite (Cr_2O_3). Laterites are classified into two types, namely saprolite with high nickel content and low nickel limonite (Makahanap and Manaf, 2010: 112).

Characterization of the research included Vickers hardness testing, endurance with ASTM E23 05 standard using CMTS model CI-30 impact testing machine, microstructure to determine the phase formed using MEIJI techno MT7100 metallurgical microscope and fracture observation using JEOL JSM-6390A SEM.

The austempering heat treatment process requires two furnaces. The first furnace serves to heat the test sample to a temperature of 950 0C and hold time for 60 minutes to obtain an even or homogeneous austenite phase. The second furnace for the austempering process using a cooling medium Zinc element which is melted at temperatures above martensite start. In this study, the temperature of austempering used was 450 0C with variations in holding the time or holding time of 30.60 and 90 minutes.

The cooling media used is liquid metal. Basically liquid metal can maintain its melting point at certain temperatures because the range of metals also has a range between the melting point and the vapor point. As used in this study, the zinc metal element (Zn) where zinc has a chemical property as follows:

1. *Molecular weight* : 65,39 g/mole
2. *Color* : Bluish-grey
3. *Melting point* : 419 0C (786.2 0F)
4. *Boiling point* : 907 0C (1664.2 0F)

3. RESULTS AND DISCUSSION

3.1. IMPACT STRENGTH

Data obtained from Charpy impact testing on variations of holding time 0 minutes, 30 minutes, 60 minutes, and 90 minutes were 209 J, 247.6 J, 194 J, and 191.3 J.

Table I. Impact Test Results

Impact Test Value, Joule (J)				
	1	2	3	Mean
Raw Material A10	210	208	0	209
Austempering 30 munite A3	254	242	247	247.6
Austempering 60 munite A6	186	192	204	194
Austempering 90 munite A9	196	192	186	191.3

Fig. 2 Impact test graph

It can be seen in the impact test results that the highest impact resistance value occurred at a time variation of 30 minutes which reached 247.6 J or increased by 18.46% for sample A0 or raw material, this was due to the finer ferrite-pearlite structure formed and proved that the transformation of the ferrite-pearlite phase into bainite has begun to appear. Whereas the material with a 60 minute holding time has decreased the impact resistance value to 194 J or a decrease of 7.17% for the raw material as well as the sample holding time of 90 minutes decreases the impact resistance value produced which reaches 191.3 J or decreases by 8, 46% of raw material. This happens because of the presence of the bainite phase which begins to form in the material with a holding time of 60 minutes and 90 minutes. Based on Figure 2, it can be seen that the longer the holding time or holding time lasts, the energy that can be absorbed by the test material decreases due to the increasing number of bainite structures formed. As stated by Reddy in his research, the longer the austempering holding time, the stronger the level of resistance due to the formation of a more homogeneous bainite structure which will cause the level of material hardness to rise and reduce the toughness of laterite steel material.

3.2. VICKERS HARDNESS

Data from the hardness test results on the austempering heat treatment process samples increased the value of the hardness that continues to increase. The data obtained from the testing of VAIC hardness in the variation of length of holding the time or holding time at 0 minutes, 30 minutes, 60 minutes, and 90 minutes were 164 HV, 177 HV, 201 HV, and 224 HV.

Table II. Results of Hardness Testing

Hardness Value, HV				
No	Sample A0	Sample A3	Sample A6	Sample A9
1	161	167	199	232
2	165	175	199	239
3	163	188	202	202
4	167	175	26	210
5	166	181	202	239
Average	164	177	201	224

Fig. 3 Graph of the results of the hardness test

The highest hardness value was generated in a variation of 90 minutes holding time which reached 224 HV, this result increased by 36.58% for raw material samples, this occurred because there was already a bainite structure formed. Where the structure of bainite has a hardness value above the ferrite-pearlite structure which on average only has a hardness value of 70 HV - 150 HV, but the hardness does not exceed the martensitic structure which can reach an average of 400 HV - 513 HV (Totten, 2006). While for material samples with a holding time variation of 60 minutes having 201 HV hardness or increasing by 14.02% on raw material samples which only have 164 HV hardness, this shows that the microstructure of bainite has begun to form with the longer holding time given. In sample A3 has a hardness of 177 HV or an increase of 7.92 raw material and still has a phase formed in A3 samples or at a 30 minute holding time which is ferrite-pearlite but has been finer and denser than raw material samples this proves that it has begun the phenomenon of the transformation of austenite into bainite. Based on Figure 3, there is an increase in the hardness of the material with the longer duration of detention, the value of the hardness increases even though it is not too significant, but it can form bainite microstructure, this can be proven from the metallographic results in Figures 4 to 7. 8 to 11 it is clearly seen that the longer the holding time, the dimple holes formed, the less it proves that the material increases the hardness value or becomes brittle. As stated by Najeeb (2014) the higher the temperature and the length of time the austempering can increase its hardness.

3.3. MICROSTRUCTURE

Analysis of data in the form of Figures 4 to 7 shows raw material samples from metallographic tests showing the raw material of laterite steel has a microstructure in the form of ferrite and pearlite, as stated by Totten (2006) that steel material with 0.2% carbon content has a ferrite microstructure perlite while the laterite steel material which is in the process of heat treatment austempering with a holding time variation has a different structure. For A3 microstructure steel specimens in the form of pearlite ferrite with fine grains then specimen A6 has a microstructure in the form of pearlite ferrite bainite matrix plate shape and specimen A9 the microstructure is a pearlite ferrite bainite matrix. Metallographic testing shows that the holding time variation in the austempering heat treatment process affects the microstructure of the steel. The longer holding time, the more bainite structure is formed.

Fig. 4 Metallographic Result A0

Fig 5 Metallographic Result A3

Fig 6 Metallographic Result A6

Fig. 7 Metallographic Result A9

Specimens with heat treatment austempering at a 30 minute holding time still have ferrite-pearlite microstructure but already have a fine grain shape it is evident that the transformation process of the austenite phase into bainite has occurred, but with a holding time of only 30 minutes phase formation is not yet clear bainite, while at holding time 60 and 90 minutes bainite microstructure has begun to be seen because the phase transformation from austenite to bainite has entered the bainite region, as in the Andrew study (2013) said the longer the holding time in the austempering process the more bainite structure formed.

From the content of Ni which has a weight percent of 1.564%, nickel which dissolves in ferrite forms an fcc unit cell (austenite stabilizer) and decreases the temperature of eutectoid (Yang, Williams, & Goldstein, 1996). Ferrite-soluble nickel increases toughness, decreases 50% ($^{\circ}\text{C}$) of Fracture Appearance Transition Temperature (FATT), even at subzero temperatures (Totten, 2007).

3.4. SEM-EDS

SEM testing aims to see fractures formed in raw materials and materials with the austempering heat treatment process.

Fig. 8 Result of Impact Fracture A0

Fig 9 Result of Impact Fracture A3

Fig. 10 Result of Impact Fracture A6

Fig. 11 Result of Impact Fracture A9

The resulting fracture in each material both raw material and after the heat treatment process is included in the type of ductile fracture, as seen from the highest hardness value obtained only 224 HV and the impact resistance value only reaches 247.6 J the value is still classified as the type of fracture resilient, can be seen from the phase formed is the bainite phase which has a type of ductile fracture, but there is no martensitic phase which has the result of brittle fracture because it has a high hardness value and low impact resistance value. Each sample provides stringent characteristics (fibrous fracture) which involve a mechanism of shifting the crystal field in a ductile metal and absorbing light and dull or dull appearance. This can be seen by the presence of dimple holes as a result of the unification process - small cavity (cavity) during loading takes place (Yuwono, 2009). As stated by Lan et al (2017) in the austempering process a bainite phase is formed which has a type of ductile fracture.

4. CONCLUSION

Based on the results of the research conducted, it can be concluded that:

1. Variation in holding time affects the mechanical properties of laterite steel. The increase in hardness in raw material from 164 HV to 224 HV or increased by

36.58% while the impact strength with the longer holding time of energy that can be absorbed by laterite steel is decreasing, ie from sample A0 which initially 209 J to 247.6 J in the sample A3, but continued to decline with the longer holding time, from A3 247.6 J to decrease to 194 J or 27.6% at A6 and continued to decline to 191.3 J in the A9 sample or 29.43%.

2. Variation in holding time affects the microstructure of laterite steel. The microstructure transformation from raw material which initially has ferrite-pearlite phase becomes changed in A3 samples which have a finer pearlite ferrite phase as well as samples A6 and A9 which begin the formation of bainite phase although there is still a pearlite ferrite phase and this can also be seen from the fracture impact test that forms a ductile fracture with dimple holes as a result of the process of uniting small cavities during loading.

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REFERENCES

- [1] ASM International. (2000). ASM Handbook. Volume 8 Mechanical Testing and Evaluation. United States of America: ASM International Handbook Committee.
- [2] ASM International. 2004. ASM Handbook. Volume 4 Metallography and Microstructures. United States of America: ASM International Handbook Committee.
- [3] Basso, D. Makahanap dan A. Manaf. 2010. Characteristics of Reduction of Laterite Iron Ore. Metallurgical Magazine Journal 25(2): 111-118.
- [4] Callister, William D. 1973. Fundamental of Material Science and Engineering. Oxford: Butterworth Heinemann.
- [5] Dieter, George E. 1986. Mechanical Metallurgy. 3rd ed. Singapore: McGraw-Hill Book Company.
- [6] Haryadi, G. D. 2006. Effect of Tempering Temperature on Hardness, Tensile Strength, and Micro Structures on Steel K-460. Jurnal Rotasi 8(2): 1-8.
- [7] Hasbi, M. Yunan, Daniel Panghuhutan Malau and Bintang Adjiantoro. 2016. Variations in Reduction of Hardness and Microstructure of Laterite Steel Through Hot Scraping. National Seminar on Science and Technology. Jakarta 2016.
- [8] Herbirowo, S., and B. Adjiantoro. 2016. Effect of Heat Treatment on Micro Structures and Mechanical Strength of Laterite Nickel Steel. Jurnal Widyariset 2(2): 153-160.
- [9] Ismoyo, A.H., Parikin, and Bandriyana. 2014. Analysis of the Effect of Hot Scraping and Forging Processes on the Mechanical Properties and Micro Structures of ZrNbMoGe Alloys. Journal Urania. 20(1): 14-21.
- [10] Murtiono, Arief. 2012. Effect of Quenching and Tempering on Hardness and Tensile Strength and Micro Medium Carbon Steel Structure for Palm Harvesters Knives. e-Dinamis Journal 2(2): 57-70.
- [11] Purwanto, H., Rudy Soenoko, Anindito Purnowidodo, and Agus Suprpto. 2015. Macro and Micro Characteristics of Steel-Aluminum Plate against Ballistic Resistance. Prosiding SNST 6th Year 2015. Faculty of Engineering, Wahid Hasyim University: Semarang (97-102).
- [12] Smallman, R.E., and R. J. Bishop. 1995. Modern Physical Metallurgy and Materials Engineering. 6th Edition. New Zealand: Reed Educational and Professional Publishing Ltd.
- [13] Sugiyono. 2012. Quantitative, Qualitative, and R & D Research Methods. Bandung: Alfabeta.
- [14] Pradia T., and Kenji Chijiwa. 2000. Metal Casting Technique. Second edition. Eighth print. Jakarta: Pradinya Paramita.
- [15] Totten, G. E. 2007. Steel Heat Treatment Metallurgy and Technologies. 2nd ed. U.S.A: Taylor & Francis Group.
- [16] Yuwono, A.H. 2009. Practical Guidebook for Material Characterization 1 Testing Damages (Destructive Testing). Jakarta: Departemen Metalurgi dan Material Universitas Indonesia.