

Study About Normalizing of AISI 1010 Steel Result Hardened by Manganese Stone Powder Using True Experimental Method

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Abstract—The study of normalization of steel by hardening using manganese powder is the design form of this study. The steel used is AISI 1020 steel because it has general standards and is suitable as a base for hardening. The interesting thing about this research is the element of manganese (Mn) in rock mineral powder as a substitute for wood charcoal which is commonly used to harden steel. Furthermore, it is known that manganese (Mn) is an important element in the chemical composition of steel which functions as a friction resistance. The explanation of this research is to describe the relationship between heat treatment, hardness, and tensile stress in steel which has undergone a hardening treatment after being normalized. The test results show that there is a strong causal relationship, the effect of temperature, and fast cooling media on tensile strength, but the temperature is not strong enough for hardness. Furthermore, SEM and EDX tests were carried out to see the topography, morphology, and chemical composition of the steel surface as a control for this relationship.

Keywords—mineral manganese powder, normalizing, causal relationship, SEM and EDX

I. INTRODUCTION

The title of the study paper on Normalization of Steel AISI 1010 Results of Manganese Powder Hardening Using the True Experimental Method originated from the desire to see the phenomenon of metal bonding at iron and manganese atoms with the help of high temperature heat. The test was carried out by hardening the steel surface using manganese mineral powder as raw material. The steel chosen is AISI 1010 standard steel. This steel classification is included in AISI (American Iron and Steel Institute) 1010, 1016, 1018, 1019, 1020 [1]. The manganese mineral used is taken from one of the mines or sources in South Central Timor Regency, East Nusa Tenggara Province. Previous research which mentioned the SEM EDX test results showed the formation of Manganese (Mn) and several other elements on the steel surface from the oxidation-reduction and pyrometallurgical processes at a temperature of 1,200 ° C [2]. From the results of these tests, the authors want to examine more deeply with other types of heat treatment such

as normalization and mechanical testing, tensile tests and hardness tests. Hardness testing is carried out because the engineering process increases hardness and toughness. Engineering that is usually done is to increase the hardness by hardening in the form of heat treatment (heat treatment) [3]. Heat treatment is often referred to as a way to increase the hardness of the material can also be used to change the useful properties or with certain interests for user needs, such as increasing flexibility, restoring elasticity after cold working.

Even heat treatment not only changes the properties of the material, but also improves the performance of the material by increasing the strength or certain characteristics of the material which has been heat-treated. Likewise, with the carburizing pack which utilizes carbon as a media for the diffusion with steel utilizing heat. Carburizing is a heat treatment process on the workpiece surface by utilizing carbon as a hardening element [4]. The addition of carbon is done by heating at a high enough temperature, namely at the temperature of the austenite in an environment containing activated carbon atoms, so that the activated carbon atoms will diffuse into the surface of the steel and reach a certain depth [5]. The working principle of this type of heat treatment is to put carbon around the workpiece when it is heated, so that the carbon will diffuse with the workpiece surface. In its implementation, because it uses manganese powder media, it is possible for the manganese reduction process to occur. There are several methods in the process of manganese metallurgy, namely pyrometallurgy and hydrometallurgy. Hydrometallurgy can be defined as a method of processing metal from rock or ore by using aqueous solvent (aqueous solution), or in detail, hydrometallurgy is a process in metallurgical work where a liquid chemical is used to dissolve a particular particle. Pyrometallurgy is a metal extraction process with heat energy, the temperature generally ranges from 500 ° C-1600 ° C and several studies using 800°C-1000 ° C [6]. Pyrometallurgy uses high temperatures to define high grade minerals. Pyrometallurgy is used by several large companies to produce commercial metals, including nickel, tin, iron and steel. Previous studies showed crushed manganese ore grains to a size of 10 mm were collected from the mine, and

then heated to 1000°C and stored for 30 minutes for high temperature treatment under N₂ protection, wherein the crystallized water and carbonate were completely decomposed [7]. The stages in the pyrometallurgical process are drying (removing water from the ore), calcination (removing crystalline water from the ore), smelting and refining (increasing the content of a metal) [8]. With the explanation above, this study aims to demonstrate steel that has received a pyrometallurgical process by heat treatment in the form of tensile testing and hardness testing after normalizing treatment.

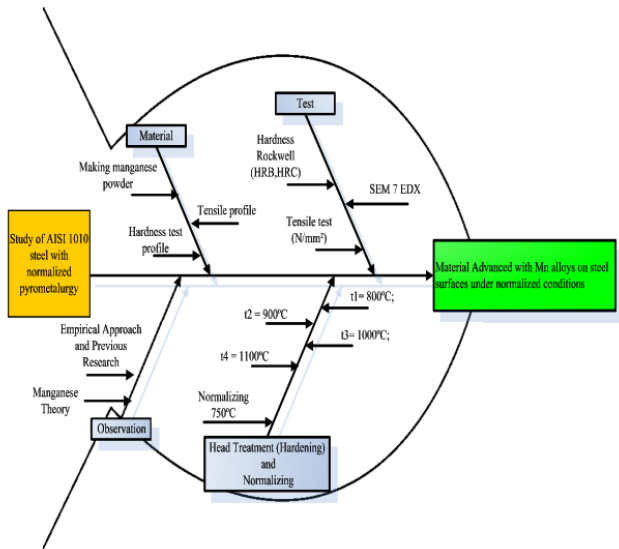


Fig. 1. Cause effect diagram.

The purpose of normalizing is to reduce residual stress, improve the mechanical properties of the steel and restore the ductility of the steel. Normalizing is a heat treatment process where the heating process reaches a temperature, then cooled slowly using an air cooling medium [9]. Furthermore, the objective of normalizing is to obtain a reliable heat-treated manganese alloy material to be used as an advanced material under normal conditions with new specifications. The material specification in question concerns tensile strength and hardness. The method used is a real experimental method that will compare the treated population with the original population. Real experimental methods are used to see the causal relationship between the population before and after treatment. Also, real experimental testing is to see the phenomenon of the causal relationship from tensile stress, hardness, and temperature variations after normalizing treatment. Further explanation of the entire study explains the research journey from input to output with a cause and effect diagram as shown in Figure 1. A Cause-and-Effect Diagram is a tool that helps identify, sort, and display possible causes of certain problems or quality characteristics.

II. METHODOLOGY

A. Research Design

- The research location was carried out at the Kupang State Polytechnic Material Testing Laboratory and the chemical composition test and Scanning Electron Microscopy (SEM) were carried out at the Materials Testing Laboratory of the Institute Teknologi Sepuluh November (ITS), Surabaya.
- The research method used is a true experimental method (true experiment design) and action for all direct observation and work on the process of making manganese powder, heat treatment and pyrometallurgical processes, normalizing, hardness testing, tensile testing, and chemical composition testing. This experimental research design is a study of possible cause and effect between the treated group and the control group (untreated) and then comparing the two.

B. Research Variable

- Independent variables: Carbon steel with heat treatment at temperatures of 800°C, 900 ° C, 1000 ° C, 1100 ° C with a grain size of 20% coal and 80 mesh and holding time 30 minutes each then normalized
- Dependent variable: Value of hardness on the HRC scale, tensile stress (N / mm²) for each temperature.
- Control variables. Chemical composition test results and Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDX).

III. DISCUSSION

A. Table of Test Results

Before carrying out the test, the treatment is what is done first. The treatment in question is ahead of the treatment process with a carburizing approach model using manganese powder and coal as a pyrometallurgical medium in oxidation. This method is done with the hope of getting an increase in the hardness and strength of the ASI 1010 steel material because of the effect of both carburizing and the addition of manganese (Mn) elements. After the heat treatment is carried out with rapid cooling, then normalizing is carried out to reduce the residual stress and the stressed steel is considered normal again. Usually normalizing is carried out at a temperature of 40-50 above the critical temperature in the Fe- C phase diagram [10].

The table 1 of test results is as follows:

TABLE I. DATA TABLE OF TENSILE AND HARDNESS TEST RESULTS

Heat Treatment	ΔL (mm)	Yield Point (N/mm ²)	ε (AL/Lo.100)	σ(F/Ao) (N/mm ²)	Violence (HRB, HRC)	
Without Heat	19,67	367,35	24,59	533,67	86,00	6,12
Heat Treatment	20,08	378,16	25,10	547,18	88,12	5,25
800°C	22,67	359,91	28,34	541,91	82,60	4,70
Water	20,81	368,47	26,01	540,92	85,57	5,36
Normalizing	16,33	479,99	20,41	705,45		21,50
800°C	16,36	486,27	21,20	721,88		19,25
Water	17,56	490,01	21,95	718,37		21,91
Normalizing	16,95	485,42	21,19	715,23		20,89
900°C	16,79	487,21	20,99	715,25		21,18
Water	17,47	499,89	21,84	714,89		20,18
Normalizing	17,03	488,76	21,29	732,67		21,88
1000°C	17,10	491,95	21,37	720,94		21,08
Water	14,87	512,45	18,59	746,01		22,67
Normalizing	15,82	506,56	19,78	735,28		21,96
1100°C	14,01	508,69	17,51	745,66		23,78
Water	14,90	509,23	18,63	742,32		22,80
Normalizing	13,78	510,88	17,23	777,34		23,24
1100°C	12,97	507,88	16,21	745,26		22,96
Water	14,66	508,25	18,33	751,44		23,72
Normalizing	13,80	509,00	17,25	758,01		23,31

B. Relationship of Heat Treatment, Hardness, and Yields Point

The data shows that in general there is an increase in tensile strength (yield point) and hardness after the entire population of the test material has been heating treated. The yield point strength is a limit where the material will continue to deform without any additional loads. The stress that causes the material to exhibit this yielding mechanism is called the yield stress. Increasing the heat treatment temperature is directly proportional to the increase in tensile strength and hardness. This can be affected by an increase in the chemical composition of carbon (C) and an increase in the chemical composition of manganese (Mn). Both of these elements are found in manganese powder and coal as objects for the high-temperature pyrometallurgy process. Elemental carbon can theoretically diffuse into iron (Fe) at austenite temperatures above 723° C. For the element manganese (Mn) in theory also increases the hardness of steel in steel. To prove an increase in manganese levels in steel, a Scanning Electron Microscope (SEM) test was carried out in the form of a scanning microscopic image magnification of 10,000 x and Energy Dispersive X-ray (EDX), namely the chemical composition of the material at each step of change based on changes in heat treatment temperature, namely from 0° C, 800° C, 900° C, 1000° C and 1100° C. A more detailed explanation of the relationship between the influence of temperature, yield point, and hardness is described descriptively through the graphic Figure 2 below.

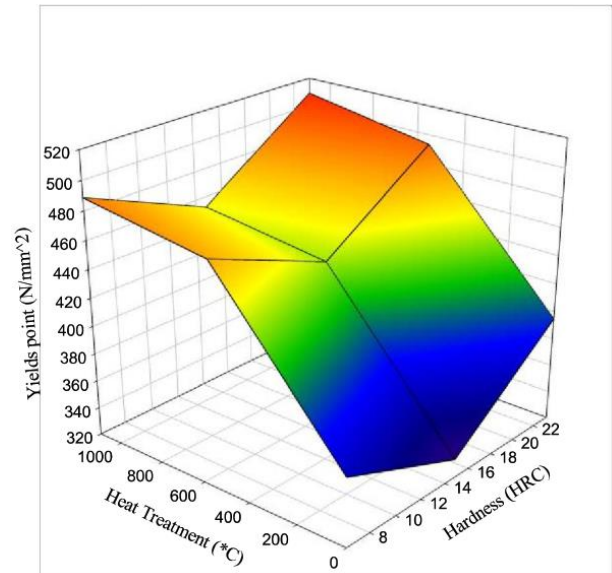


Fig. 2. Graph of relationship of heat treatment, hardness, and Yields point.

C. The Mathematical Model of Tensile Strength

Tensile testing uses standards by international standards, namely ASTM E 8 and ASTM E 8M by American standards, and if in Japan it uses JIS 2241 standards [11].

Value of R or Multiple R. shows the correlation between the independent variable and the dependent variable (not independent). The R-value of 0.8415 shows the correlation value of the variable X (temperature) with the variable Y (Tensile Strength) (Table 2). Furthermore, R Square is the coefficient of determination which shows the direct effect of the independent variable on the dependent variable which is expressed as a percentage. The determination coefficient of 0.7082 means that the variable X (temperature) directly affects the variable Y (tensile strength) by 70.82% while (100-70.62) % = 29.38% is influenced by other factors outside the variable X. ANOVA test results show: Adjusted R Square is the coefficient of determination that has been corrected for the number of variables and the sample size so that it can reduce the element of bias in the event of additional variables. Adjusted R Square of 0.6920 means that the variation of variable Y can be explained by variable X of 69.20% or variable X (temperature) affects variable Y by 69.20%. From the above results, the regression equation $Y = 569,3343 + 0.1833 X$ is obtained, which means that if the variable X is in the 0 point position, the value of the Y variable is 569.3333 (no change/stagnation) and if the variable X experiences an increase / decreasing, the variable Y follows the algebraic operation formed from the regression equation. The resulting regression equation is not a very ideal relationship because there is still bias due to the influence of other factors, but at least it reflects the desired holding time relationship to the yielding point strength.

TABLE II. DATA TABLE OF TENSILE RESULTS

R Estimate	Rsqr	Adj Rsqr	Standard Error of	
0,8415	0,7082	0,6920	45,0590	
Coefficient	Std. Error	t	P	
y0	569,3343	21,5836	26,3781	<0,0001
a	0,1833	0,0277	6,6090	<0,0001
Analysis of Variance:				
	DF	SS	MS	
Regression	2	976241,3732	4881320,6866	
Residual	18	36545,6698	2030,3150	
Total	20	9799187,0430	489959,3521	
Corrected for the mean of the observations:				
	DF	SS	MS	
Regression	1	88681,4881	88681,4881	
Residual	18	36545,6698	2030,3150	
Total	19	125227,1579	6590,9030	

TABLE III. DATA TABLE OF HARDNESS TEST RESULTS

R Estimate	Rsqr	Adj Rsqr	Standard Error of	
0,5701	0,3250	0,2875	325,9755	
Coefficient	Std. Error	t	P	
y0	467,6013	115,4572	4,0500	0,0008
a	-0,4587	0,1558	-2,9442	0,0087
Analysis of Variance:				
	DF	SS	MS	
Regression	2	1753236,9157	876618,4578	
Residual	18	1912680,4427	106260,0246	
Total	20	3665917,3584	183925,8679	
Corrected for the mean of the observations:				
	DF	SS	MS	
Regression	1	921104,5851	921104,581	
Residual	18	1912680,4427	106260,0246	
Total	19	2833785,0278	149146,5804	

D. Hardness test Math Model

Value of R or Multiple R, shows the correlation between the independent variable and the dependent variable (not independent). The R-value of 0.5701 shows the correlation value of the X variable (head treatment and normalizing) with the Y variable (hardness) (Table 3). Furthermore, the value of R Square is the coefficient of determination which shows the direct effect of the independent variable on the dependent variable which is expressed as a percentage. The determination coefficient of 0.3250 means that variable X directly affects variable Y (hardness) by 32.50% while (100-32.50)% = 67.5% is influenced by other factors outside variable X. Adjusted R Square is the coefficient of determination that has been corrected by the number of variables and sample size to reduce the element of bias if there is an addition of variables. Adjusted R Square of 0.2875 means that the variation of the Y variable (hardness) can be explained by the X variable of 28.75% or the X variable (holding time) affecting the Y variable by 28.75%. From the results above, the regression equation $Y = 467.60 + (-0.48587)X$ is obtained, which means that if the variable X is in the 0 point position, the value of the Y variable is 467.60 (which means that it is very unstable and changes from the value of violence, which should be.) and if the variable X experiences an increase/decrease, the variable Y even changes but does not follow the algebraic operation formed from the regression equation. In other words, the change in heat treatment temperature does not have a significant effect on hardness.

E. Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray analysis (EDX)

Scanning Electron Microscopy (SEM) is a high-resolution microscope that utilizes a high-energy electron beam to examine objects on a very small scale. Observations provide information regarding topography and morphology. Meanwhile, the composition of elements and compounds along with their respective relative amounts uses EDX (Energy Dispersive X-Ray analysis).

TABLE IV. CHEMICAL COMPOSITION TEST RESULTS ON THE ORIGINAL MATERIAL WITH EDX

E1	AN	Series	unn	C.nor	C.Atom	C.Error
			(wt.%)	(wt.%)	at. %	%
Fe	26	K. Series	95,98	93,10	80,53	4,40
C	6	K. Series	2,86	2,77	11,16	0,50
O	8	K. Series	2,26	2,19	6,61	0,40
Mn	25	K. Series	2,00	1,94	1,71	0,20
			Total	103,10	100,00	100,00

Figure 3 shows the flat topography of the surface of the original AISI 1010 carbon steel object. The shape of the surface, in general, has not been influenced by heat treatment and other elements. The surface morphology in the image shows the flat surface. The diameter or length of each atom is 1-2 mm, 10 mm, and 100 mm with smooth characteristics and a very flat surface. In the celebration image, you can see the colors of each element into one compound. The celebration image of the AISI 1010 material is clarified by the results of the chemical composition test in Table 4, namely 4.4% Fe, 0.5% C, and 0.2% Mn at C Error.

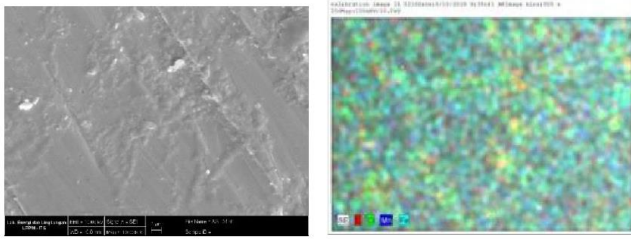


Fig. 3. SEM test results of original materials with calibration images.

According to the results of the previous ANOVA discussion, it was stated that temperature did not significantly affect the hardness of the material. To answer this relationship, in this test a control test was carried out with SEM and EDX. In SEM and EDX testing for materials that are heat-treated at a temperature of about 950 ° C austenite with air and water cooling, then normalizing is carried out. Both types of coolants are used to control the characteristics that occurred in the previous test (Table 5).

TABLE V. THE CHEMICAL COMPOSITION OF THE INGREDIENTS AIR-COOLED HEAT TREATMENT

E1	AN	Series	unn (wt.%)	C.nor (wt.%)	C.Atom at.%	C.Error %
Mn	25	K. Series	37,22	38,44	21,92	1,60
Fe	26	K. Series	32,32	33,38	18,72	1,60
O	8	K. Series	20,13	20,79	40,70	2,40
C	6	K. Series	6,76	6,98	18,20	0,90
Si	14	K. Series	0,39	0,40	0,44	0,00
Al	13	K. Series	0,01	0,01	0,01	0,60
		Total	96,83	100,00	100,00	

The heat treatment test with air conditioning was then normalized as shown in Figure 4, showing that the surface topography of the material is wavier, slightly rough, it seems to blend with the original material. Both 1-2 mm, 10 mm, and 100 mm atomic size shots show the same characteristics. Because the heat treatment approach is carried out with the help of manganese powder as a pyrometallurgical medium, it can be seen that the influence of other elements on the surface of AISI 1010 steel. The celebration image shows the difference with the original material. The elements Mn, Si, and Al are transformed on the surface of the object. The morphology of the surface of an object that has been influenced by other elements shows the rough atoms shrouded in the surface of the object. This is clearer for pictures with a size of 1-2 mm. From the table, the results of the material composition test show the addition of 1.6% Mn, 0.6% Al and the addition of% Carbon from 0.5% to 0.9%. Further research is needed on this phenomenon whether there is total diffusion with chemical bonds or just sticking to the steel surface.

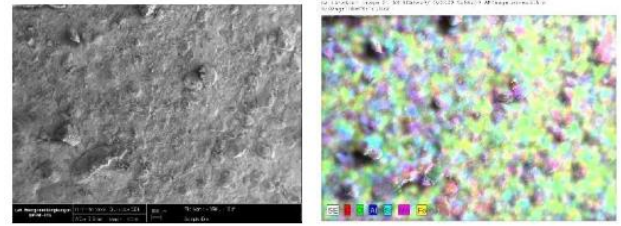


Fig. 4. SEM test results on materials that have been heating treated and quenched with air and a celebration image.

Likewise, with the heat treatment with water cooling, it appears that there is an effect of adding Mn and Si elements. Mn content was higher than heat treatment with air cooling by 2.3% (Table 6). This shows that the type of quenching has a significant effect on the chemical composition of the material. The heating treated with water-cooled normalized as shown in Figure 5.

TABLE VI. CHEMICAL COMPOSITION OF WATER-COOLED HEAT TREATMENT MATERIALS

E1	AN	Series	unn (wt.%)	C.nor (wt.%)	C.Atom at.%	C.Error %
Mn	25	K. Series	54,16	53,71	32,84	2,30
O	8	K. Series	22,33	22,15	46,50	2,70
Fe	26	K. Series	20,41	20,24	12,17	1,10
C	6	K. Series	2,40	2,38	6,65	0,40
Si	14	K. Series	1,34	1,33	1,59	0,10
Al	13	K. Series	0,20	0,20	0,25	0,00
		Total	96,83	100,00	100,00	

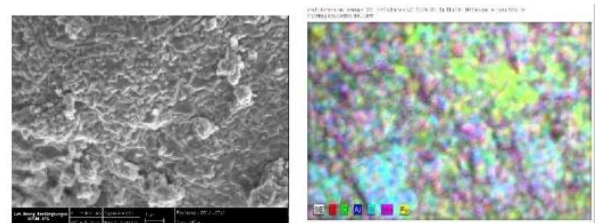


Fig. 5. SEM test results on materials that have been heating treated with water-cooled and celebration image.

Both topography and morphology show that the surface of objects is wavier, rougher and there are rough bonds that occur on the surface of AISI 1010 steel. Images with large and small sizes show this phenomenon. The descriptive explanation can also be seen from the calibration image where the Mn crystals have more pink color than the pink color in the celebrated image of the original object.

IV. CONCLUSION

- Although there is a causal relationship between the increases and is directly proportional to the increase in temperature and yield point, temperature does not have a significant effect on hardness in this study.
- The data show that anyone knows that there is a strong difference between treatment temperature change and

normalization with tensile strength. This is due to the treatment of rapid immersion resulting in changes in chemical composition with the addition of carbon (C) and Manganese (Mn) as evidenced by the results of material composition testing.

- SEM and EDX testing on the surface of objects as a control of the phenomenon of the causal relationship between temperature, hardness and stress, it can be seen that there are other elements in the hardening process using manganese powder. The chemical composition addition elements in question are Manganese (Mn) and Silicon (Si).
- Further testing is needed to determine whether the chemical bonds that occur on the steel surface move into the material by diffusion or not and examine what chemical elements make up it.

REFERENCES

- [1] K. Bimariga and L. Noerochiem, "Pengaruh Variasi Kuat Arus Terhadap Ketebalan, Kekerasan dan Ketahanan Korosi Hasil Elektroplating Nikel-Hard Chromium Pada Baja AISI 4340," *J. Tek. ITS*, vol. 8, no. 1, 2019.
- [2] A. Wibawa, "Pengaruh Temperatur Terhadap Reduksi Bijih Mangan," pp. 221–228, 2014.
- [3] P. Biswas, A. Kundu, D. Mondal, and P. Kumar Bardhan, "Effect of heat treatment on microstructure behavior and hardness of en 8 steel," *IOP Conf. Ser. Mater. Sci. Eng.*, vol. 377, no. 1, 2018.
- [4] K.S. Hassan, "Comparative of wear resistance of low carbon steel pack carburizing using different media," *Int. J. Eng. Technol.*, vol. 4, no. 1, p. 71, 2015.
- [5] M. Dwiharsanti, W.S. Jaman, and S. Virdhian, "Perancangan Eksperimen Baja Karbon Rendah Hasil Proses Pack Carburizing Dengan Metode Eksperimen Faktorial," *Journal of Industrial Research (Jurnal Riset Industri)*, vol. 10, no. 2, pp. 92-97, 2016.
- [6] R.H. Pangaribuan, J. Patrick, A.B. Prasetyo, A. Maksum, B. Munir, and J.W. Soedarsono, "The effect of NaOH (natrium hydroxide) to slag nickel pyrometallurgy in different temperature and additive ratio," *E3S Web Conf.*, vol. 67, no. January, 2018.
- [7] B. Zhang and Z.-L. Xue, "Kinetics Analyzing of Direction Reduction on Manganese Ore Pellets Containing Carbon," *Int. J. Nonferrous Metall.*, vol. 02, no. 03, pp. 116–120, 2013.
- [8] S. Taruminkeng, E.J. Mustopa, and L. Hendrajaya, "Termodinamika Dalam Memahami Proses Pengolahan Mineral," In *Prosiding Seminar Nasional Fisika (E-Journal) Vol. 5*, pp. SNF2016-ERE, 2016
- [9] A.S. Nugroho, G.D. Haryadi, and A.T. Hardjuno, "Pengaruh Proses Normalizing terhadap Nilai Kekerasan dan Struktur Mikro pada Sambungan Las Thermite Baja NP-42," *Jurnal Teknik Mesin*, vol. 2, no. 3, pp. 249-257, 2014.
- [10] O.R. Adetunji, P.O. Aiyedun, S.O. Ismaila, and M.J. Alao, "Effect of Normalizing and Hardening on Mechanical Properties of Spring," *J. Miner. Mater. Charact. Eng.*, vol. 11, no. 08, pp. 832–835, 2012.
- [11] ASTM E18/18M-11, "Standard Test Methods for Rockwell Hardness of Metallic Materials," *ASTM Int.*, p. 38, 2018.