Factorial Identification on Surface Hardness of Cut Low Carbonsteel with Hardening Process using Manganese Minerals Powder

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Abstract: This study wanted to ensure the diffusion of manganese on the surface of the carbon steel cleavage into the steel core using high heat with a hardening process and a pack carburizing approach. The purpose of the pack carburizing process approach is the hardening of the steel surface at high temperatures using manganese rock powder mineral as a substitute for carbon such as solid charcoal. Previous research showed that the chemical composition test formed elemental Manganese (Mn) and several other elements on the steel surface from the pyrometallurgical oxidation and reduction process of manganese rock powder at a temperature of 1000 °C. The purpose of this test is to determine the factorial effect of steel cleavage surface hardness with factors, temperature, coal composition, and holding time. Control of chemical composition by using Energy Dispersive X-ray (EDX). The ANOVA test results show that the percentage of coal has a significant effect on increasing the hardness of the test sample. Control of chemical composition showed that there was an atomic diffusion process with increasing levels of carbon (C), Manganese (Mn) and other elements, Silicon (Si), Aluminum (Al). EDX data information explains that to form a hard steel surface not only by using carbon but also with manganese rock powder minerals.

1 INTRODUCTION

Developers and seekers of mineral alloy steel will be interested in discussing the manganese mineral found on the island of Timor. Not because of the abundant minerals but also the high content of manganese (Mn). It is known as one of the best manganese rock minerals with an Mn content of around 20% -5% (Panjaitan, 2011). One of these mining locations is the Koa area and its surroundings in the Mollo Barat District, Timor Tengah Selatan Regency (TTS) with a total deposit of 454,123,065.8 m³ (Harjanto, 2011). There did two types of manganese ores found in the area, namely manganese layers and manganese nodules (Idrus *et al.*, 2013). Manganese minerals did widely distributed in various forms such as oxides, silicates, carbonates of the most common compounds. Both types of manganese minerals can be able formed into powder for this research. In addition to other technical functions, the function of manganese in steel is as a special alloy that causes steel to be friction-resistant, impact-resistant, and has high hardness. That is why manganese is so important in the metal or machinery industry. Accordingly, the manufacturing industry is very interested in developing new types of technological equipment that allow the application of methods to modify the surface of parts by processing them with lower resources (Skeeba, Ivancivsky and Martyushev, 2021). Previous studies by researchers tried to link manganese rock powder and high heat. This research is to ensure the presence of metal between iron and manganese atoms on the surface of carbon steel with the help of high-temperature heat with pack carburizing and pyrometallurgical approaches. Pack Carburizing is a form of hardening of steel using a medium containing carbon at a temperature of 800 - 950 °C then quenching quickly. For example, ordinary gears use AlNas embedding particles, whose carburizing temperature is in the range of 930–980 °C(Liu *et al.*, 2021). At that temperature in an environment containing activated carbon atoms, activated carbon atoms will diffuse to the surface of the steel and reach a certain depth (Mirantie Dwiharsanti, 2016). Other tests concluded that the optimumaustenitizing temperature is about 840◦C for the test steel(Chen *et al.*, 2021). Pyrometallurgy is a metal extraction process with heat energy at a general temperature used in the range of 500 °C-1600 °C (Wibawa, 2018).

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Previous research with Energy Dispersive X-ray (EDX) showed the formation of Manganese (Mn) and several other elements on the surface of steel samples in the oxidation-reduction process at a temperature of 1000 °C (Rerung, Sapar and Dimu, 2019). Next, identify the factors that affect the hardnessof the steel cleavage after receiving the previous hardening treatment. The purpose of testing steel pieces is to analyze effect of hardness from the steel surface towards the steel core. The control variable remains using the chemical composition with EDX. The results test will be important to answer industry needs regarding surface hardening and steel resistance to friction. The addition of Manganese (Mn) will make the steel test sample hard, friction-resistant and high-strength. (Bleck and Haase, 2019). The usefulness of the processed steel is to produce steel that can work on various special equipment such as attack-resistant excavator bucket teeth (Winarto *et al.*, 2019).

The development of heavy-duty manufacturing technology such as automotive and heavy equipment has driven the engineering of Advanced High Strength Steels (AHSS), namely high-strength manganese alloy steels (SimoneKaar 1,*, DanielKrizan 2, ReinholdSchneider 1, 2019). The description of the AHSS data starts from traditional high-strength steels which include manganese carbon steel (C-Mn), hardened steel, high-strength free intergroup steel (HSS-IF), and high-strength low-alloy steel (HSLA). . The concept of Advanced High Strength Steel (AHSS) had divided into high strength steel into conventional HSS and advanced high strength steel. The strength of AHSS ranges from 500 Mpa to 1500 Mpa as and one of the techniques used to achieve AHSS is to increase the hardness or tensile stress using hardening in the form of heat treatment (Herwandi, 2005).

The next research step is to make a factorial design. The design of factorial experiments is a procedure for placing treatments into experimental units with the main objective of obtaining data that meet scientific requirements. Thus research can be measured and strict control inprocesses and methods.

2 METHODOLOGY

This research is a continuation of previous research that focuses on the interaction of manganese and iron on the surface of carbon steel with a hardening approach with pack carburizing and pyrometallurgy. Furthermore, this study will analyze the diffusion of manganese from the steel surface into the steel core by measuring the hardness and analyzing the chemical composition of each element. Measuring the hardness of the cut part of the test sample by selecting the position of the outermost part of the surface of the steel shell. The type of penetration that has been used is Rockwell B with a spherical penetration measuring 1/16". Furthermore, testing the chemical composition at these points. This is to ensure the influence of manganese in manganese powder on the hardness of carbon steel.

2.1 Flow Chart Diagram

Experimental research tries to explain the benefits of manganese on the island of Timor (East Nusa Tenggara) in this study. Another important thing is the workflow chart from sampling, the methods, to experimental processes. The description can be illustrating in a research flow chart. Furthermore, developing the research with critical thinking with statistical rules to help find the factors that affect the hardness of the test sample. Many experiments involve two or more the factors. This concept creates a factorial design in which every possible level of a combination of all the factors will be available. (Salomon et al., 2017). The design process will place independent variables with two different levels namely temperature, holding time, and percentage of coal catalyst. The dependent variable design is the hardness value. Factorial designs design concurrent trials of two or more single trials with two factors often found in multilocation trials (Lawson, 2016). To further elaborate the research steps as shown in the flow chart in Figure 1 below.

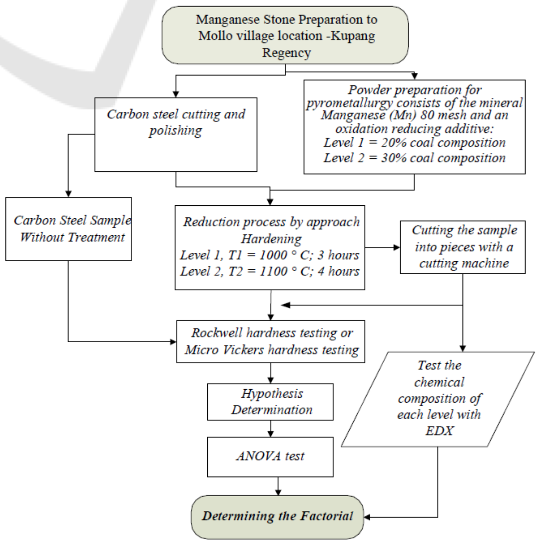

Figure 1: Research flow chart.

2.2 Research Roadmap

Further explanation of the research explains the research journey from input to output with a cause-and-effect diagram. Cause-and-Effect Diagram is a research Road Map tool that helps identify, sort, and display the possible causes of a certain quality or characteristic. (Deshpande, 2008). The diagram shows the whole concept of thinking by connecting theory with several methods. These methods are experimental and statistical with tentative assumptions. The diagram picture will describe the research journey from the beginning to the end of the study. The information displayed is time, process, cause and effect relationship, and achievement.

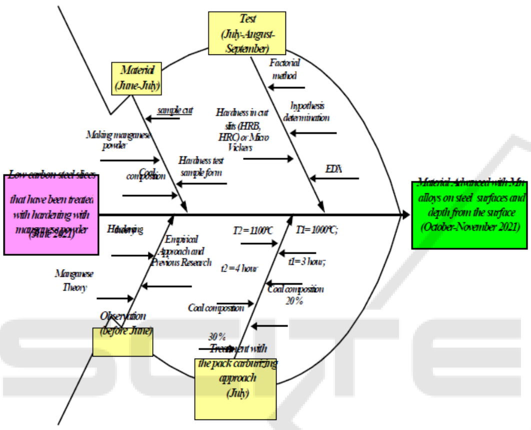

Figure 2: Road Map with Cause Effect Diagram.

2.3 Research Design

2.3.1 Research Site

The location was carried out at the Kupang State Polytechnic Materials Testing Laboratory and testing of chemical composition and Energy Dispersive X-ray (EDX) was carried out at the Materials Testing Laboratory of the Tenth Institute of Technology (ITS) November Surabaya.

2.3.2 Research Methods

Experimental research may be of three types; Pre experiment, Quasi experiment and True experiment. All these types have contrastive characteristics (Qasim, Imtiaz and Alvi, 2014). Study, experimental, or research design is the backbone of good research. It directs the experiment by orchestrating data collection, defines the statistical analysis of the resultant data, and guides the interpretation of the results (Knight, 2010). The experimental design of the action starts from the preparation of materials, namely the manufacture of manganese powder, the manufacture of coal powder, the manufacture of heat treatment boxes, and the cutting of test samples. All processes take place with the team by working directly in the Kupang State Polytechnic Material Testing Laboratory. The next step is to conduct an experiment using the true experiment method, namely a study of the possibility of causality between the treated group and the untreated control group and compare the two. The experiment in question is hardening with pack carburizing and pyrometallurgical processes. The last step is to carry out hardness testing and chemical composition testing with EDX. To explain the factors that affect the hardness of materials and their interactions using the factorial experimental method. Factorial Experimental Method is an experiment that combines or crosses all certain the factors with each other at the factor level in the experiments. Factorial identification will look at the influence of individual factor and the interaction of the factors on the measurement of the target object in the study. Determination of significance with going test the significant level of the influence of the factors and their interactions using the ANOVA method. The ANOVA test used one-way ANOVA with a significance level (α) = 5%. If F-count < F-table, then the null hypothesis is accepted and, the first hypothesis was rejected and, if F-count > F-table, then the null hypothesis is rejected and, the first hypothesis is accepted.

2.3.3 Research Variable

Factorial design is probably the most powerful statistical technique for research into any manufacturing process for the purpose of quality improvement (Teow, 2005). This article discusses the practical aspects of using a full factorial design optimization of heat treatment variables to increase the cleavage hardness of carbon steel. Variables The research variables are arranged as follows:
Independent Variables:
Level 1: Hardening temperature T1 = 1000ºC with 20% coal and holding time 3 hours Level 2: Hardening temperature T2 = 1100ºC with 30% coal and holding time 4 hours –
Dependent variable:
Hardness value with Rockwell Hardness on the HRC scale.
Control variable
Chemical composition test results and Scanning Electron Microscopy (SEM)

3 DISCUSSION

Researchers arrange relationships between variables according to the concept of causal relationships in the previous section. If one independent variable affects the dependent variable, it is call a one-factor experiment because there is only one modifier. This kind of relationship is a simple relationship between variables. The causal relationship will be more complicated if it is a combination of several factors. We can say that factorial is an experiment consisting of two or more independent variables. Two independent variables that influence is called a two-factor factorial, and if three independent variables affect it is called a three-factor factorial and so on. In this study, three independent factors are considered to have an effect on the hardness of carbon steel after hardening treatment, namely temperature (T), holding time (t), and coal percentage C (%). have been carried out are as follows:

3.1 Experiment Planning

The response variable is the hardness of the low carbon steel piece hardened by the pack carburizing approach. The Factors using three independent variables, namely temperature holding time and coal composition. A process is also a form of pyrometallurgical approach process. Each factor has two levels with austenitizing temperatures consist of 1000 °C and 1100 °C and holding time factors of 3 hours and 4 hours, respectively. The carburizing medium uses manganese powder with a coal percentage of 20% and 30%.

The experimental design table format and the table of hardness test results on treated carbon steel in the form of a matrix are as follows:

Table 1: Experimental Design.

Independent Variables:	Level	
Hardening temperature (T)	1000°C	1100°C
Holding Time (t)	3 hour	4 hour
Coal Percentage (C)	20 %	30%

One of the reasons for using the table format is because in carrying out heat treatment tests where the temperature requires variations in holding time. Although the format of the relationship between these variables is quite complex and may not be common, this study tries to answer the influence of these factors on the sample test.

3.2 Experiment Execution

In general, pyrometallurgical and hydrometallurgical processes are two technologies for extracting and separating metals from minerals to produce refined metals. Pyrometallurgy is a process that utilizes high temperatures to chemically change minerals, separating the desired metal from other materials and ultimately reducing metal oxides to free metals (Sara Yasipourtehrani, Vladimir Strezov*, Tim Evans, 2020). The experimental implementation of hardening and pyrometallurgical processes with a pack carburizing approach on low carbon steel is follow the experimental design. The entire test takes place under strict measurement controls to obtain accurate data. If you pay attention to the table of hardness results, it can be see that there is a distribution of data between the hardness of 95.44 HRB - 106.12 HRB. The data shows there is an increase in hardness from the original sample, namely low carbon steel with a hardness ranging from 180 HB ~ 89 HRB. Support theory is the Fe - C phase diagram at austenite temperatures above 800 C which shows a diffusion process in carbon steel. Two elements of the diffusion process into hope are elements of Carbon (C) and elements of Manganese (Mn). This provisional assumption will be meaningful in engineering the properties of steel to become friction-resistant steel. Friction-resistant steel is a special steel that is rich in the element Manganese. The hardness results from the pack carburizing process can be seen in table 2.

Table 2: Hardness Test Results table.

Treatment			Hardness (HRB)			
Temperature (T)			1000 °C (T1)		1100 °C (T2)	
Holding Time (t)			3 hours (t1)	4 hours (H2)	3 hours (t1)	4 hours (t2)
Treatment Interaction			T1t1	T1t2	T2t1	T2t2
	20% (C1)	1	102,77	96,66	97,69	103,30
		2	97,30	102,55	102,10	99,36
		3	95,44	102,66	96,46	101,79
coal		4	100,05	99,67	100,12	99,65
composition to		5	99,80	98,93	99,80	102,89
manganese	30% (C2)	1	103,90	105,55	104,88	106,12
powder		2	98,77	99,01	103,44	104,90
		3	105,02	103,55	105,77	98,64
		4	103,11	105,77	98,99	104,25
		5	102,00	100,82	104,50	105,22

3.3 Determination of Hypothesis

H01: There is no significant difference between the temperature factors in influencing the hardness of carbon steel

H11: There is a significant difference between the temperature factors in influencing the hardness of carbon steel.
H02: There is no significant difference between holding time factors in influencing the hardness of low carbon steel.
H12: There is a significant difference between the holding time factors in influencing the hardness of carbon steel.
H03: There is no significant difference between the percentage of coal factors in influencing the hardness of carbon steel.
H13: There is a significant difference between the percentage of coal factors in influencing the hardness of carbon steel.
H04: There is no significant difference between the interaction of temperature and holding time factors in influencing the hardness of carbon steel.
H14: There is a significant difference between the interaction of temperature and holding time factors in influencing the hardness of carbon steel.
H05: There is no significant difference between the interaction of temperature factor and coal percentage factor in influencing the hardness of carbon steel.
H15: There is a significant difference between the interaction of temperature factor and coal percentage factor in influencing the hardness of carbon steel.
H06: There is no significant difference between the interaction of holding time factor and coal percentage factor in influencing the hardness of carbon steel.
H16: There is a significant difference between the interaction of holding time and percentage of coal in influencing the hardness of carbon steel.
H07: There is no significant difference between the interaction of temperature factor, holding time and coal percentage factor in influencing the hardness of carbon steel.
H17: There is a significant difference between the interaction of temperature factors, holding time and coal percentage factors in influencing the hardness of carbon steel.

3.4 ANOVA Test

The basis of the experimental trial is a design matrix with a factorial design. Development of a significant regression model of process parameters to predict quality characteristics by Design Of Experiment (DOE) (Chauhan et al., 2017). The first step is to do the Normality test before doing the ANOVA test. The normality test aims to determine: "Is the data presented is normally distributed or not?". The test results show a p-value > 0.1 or greater than the 0.05 significance level, then the data can be the key to the normal distribution as shown in Figure 3. Next, perform an ANOVA test on the hardness value of the test sample steel.

The ANOVA test results in the ANOVA table provide information about three variables as sources of variation and their interactions that affect steel hardness. A more in-depth explanation with ANOVA numbers are degrees of freedom (df), the sum of squares (SS), mean square (MS), F-count, and F-table.

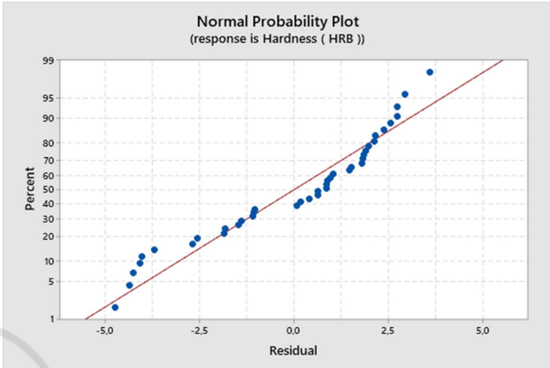

Figure 3: Normal Probability Plot.

This test will be more interesting by integrating experimental design and factorial testing that can scientifically produce mathematical equations. Then control it by displaying the results of the chemical composition test on the hardening steel section as a test sample.

Furthermore, it will be equipped with more detailed and complete data and discussions.

The results of the OF Variance Analysis (ANOVA) test show that coal has a very dominant effect on the hardness of steel that has been treated compared to temperature and holding time or its interactions.

Table 3: ANOVA test.

Source	DF	Adj SS	Adj MS	F-Value	F-Table	P-Value
C (C1;C2)	1	106,341	106,341	17,37	4,1	0
T (T1;T2)°C	1	9,39	9,39	1,53	4,2	0,224
C (C1;C2)*T(T1;T2)°C	1	3,894	3,894	0,64	4,2	0,43
Error	36	220,398	6,122			
Total	39	340,023				

The effect of coal causes an increase in hardness in the approach of the pack carburizing process because coal contains a lot of carbon. Carbon is one of the elements that increase the hardness of steel. The treatment temperature above the austenite temperature in the iron-carbon phase diagram generally has no significant effect on the test model. An interesting thing for the study is: "What is the role of manganese (Mn) or manganese rock powder in the process of increasing the hardness of steel?". To explain it, let's look at the results of the following chemical composition test: formed not only with the elements but also with manganese rock powder in hardening and pyrometallurgical processes.

3.5 Chemical Composition Test

Chemical Test is useful to support hardness test results. The chemical composition will provide information about: "How Manganese can diffuse into the steel cleavage. It this in line with the expectations of the research design. Besides Manganese, another b important element is Carbon (C) for steel hardening. Table 5 shows the hardness of the Mn diffusion process. which occurs on a small scale of about 1% around the point of intersection of carbon steel. In addition to the elements Mn, there are also elements of Carbon (C), Silicon (Si), and Aluminum (Al). Table 4 is the chemical composition of the original sample of carbon steel shows that no there is an element of Mn and other additives.

Table 4: Low Carbon Steel Chemical Composition.

El	AN	Series	unn.C	norm. C	CAtom	CError
			(wt.%)	(wt.%)	(at.%)	(%)
Fe	26	K-Series	87,36	98,47	93,27	4,1
C	6	K-Series	1,36	1,53	6,73	0,1
Total:			88,72	100,00	100,00	

Table 5: Chemical composition of treated carbon steel cut cleavage.

El	AN	Series	unn.C	norm. C	CAtom	CError
			(wt.%)	(wt.%)	(at.%)	(%)
Mn	25	K-Series	24,19	25,30	14,25	1,04
Fe	26	K-Series	55,50	60,79	44,14	2,13
O	8	K-Series	8,30	8,55	28,80	2,88
C	6	K-Series	4,06	4,19	12,60	0,54
Si	14	K-Series	0,15	1,16	0,20	0,00
Al	13	K-Series	0,01	0,01	0,01	0,00
Total:			92,21	100,00	100,00	

Observing the treatment gives a strong impression that the hardening process is going well. The diffusion process into the steel cleavage is not only the C element but the Mn element. The Mn element was importanted in the engineering of specialed steel materials with the ability to withstand friction and withstand impact.

Supporting data from the carbon composition test results found that pursuing steel surfaces can be formed not only with the elements but also with manganese rock powder in hardening and pyrometallurgical processes.

4 CONCLUSIONS

After conducting research and discussion resulted in several important things as conclusions, namely:

- Hardening factorial design in hardening and pyrometallurgical processes shows that coal composition, temperature, and holding time factors increase the hardness of the carbon steel cleavage sheath.
- The most dominant factor in increasing the hardness of steel is the percentage of coal composition. Coal is a solid mineral that is rich in carbon.
- Chemical composition test results show the formation of Manganese (Mn), Carbon (C), in small amounts of silicon (Si), and Aluminum (Al) in the steel cleavage sheath.
- The general explanation of this research is that the steel hardening process by pyrometallurgy shows an increase in the hardness of carbon steel not only because of the content ofCarbon (C) but also the element Manganese (C) as one of the constituents of alloy steel strength. Manganese alloy steel is useful for spare parts for heavy work industrial equipment such as crushers, excavator buckets, wheel loaders, marine ship plates, sprockets and so on.

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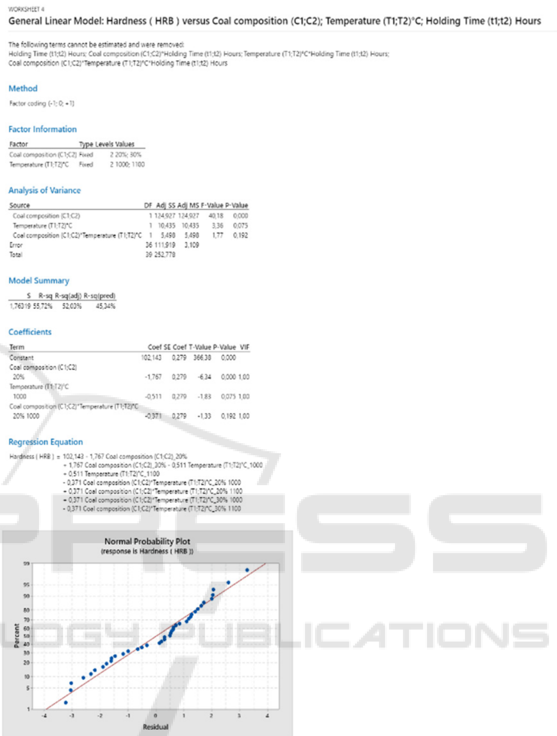
APPENDIX


WORKSHEET 4

General Linear Model: Hardness (HRB) versus Coal composition (C1;C2); Temperature (T1;T2)°C; Holding Time (t1;t2) Hours

The following terms cannot be estimated and were removed:
Holding Time (t1;t2) Hours; Coal composition (C1;C2)*Holding Time (t1;t2) Hours; Temperature (T1;T2)°C*Holding Time (t1;t2) Hours;
Coal composition (C1;C2)*Temperature (T1;T2)°C*Holding Time (t1;t2) Hours

Method

Factor coding (-1; 0; +1)

Factor Information

Factor	Type	Levels	Values
Coal composition (C1;C2)	Fixed	2	20%; 30%
Temperature (T1;T2)°C	Fixed	2	1000; 1100

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Coal composition (C1;C2)	1	124,927	124,927	40,18	0,000
Temperature (T1;T2)°C	1	10,435	10,435	3,36	0,075
Coal composition (C1;C2)*Temperature (T1;T2)°C	1	5,498	5,498	1,77	0,192
Error	36	111,919	3,109		
Total	39	252,778			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1,76319	55,72%	52,03%	45,34%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	102,143	0,279	366,38	0,000	
Coal composition (C1;C2)					
20%	-1,767	0,279	-6,34	0,000	1,00
Temperature (T1;T2)°C					
1000	-0,511	0,279	-1,83	0,075	1,00
Coal composition (C1;C2)*Temperature (T1;T2)°C					
20% 1000	-0,371	0,279	-1,33	0,192	1,00

Regression Equation

Hardness (HRB) = 102,143 - 1,767 Coal composition (C1;C2)_20%
+ 1,767 Coal composition (C1;C2)_30% - 0,511 Temperature (T1;T2)°C_1000
+ 0,511 Temperature (T1;T2)°C_1100
- 0,371 Coal composition (C1;C2)*Temperature (T1;T2)°C_20% 1000
+ 0,371 Coal composition (C1;C2)*Temperature (T1;T2)°C_20% 1100
+ 0,371 Coal composition (C1;C2)*Temperature (T1;T2)°C_30% 1000
- 0,371 Coal composition (C1;C2)*Temperature (T1;T2)°C_30% 1100