ARGON FLOW RATE EFFECT ON HARDNESS OF CARBON THIN FILMS ON SKD11 STEEL USING TARGET MATERIAL FROM BATTERY CARBON RODS WITH PLASMA SPUTTERING METHOD

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3. Reviewer (25 April 2020)
4. Payment journal (28 April 2020)
5. Publish (30 Mei 2020)

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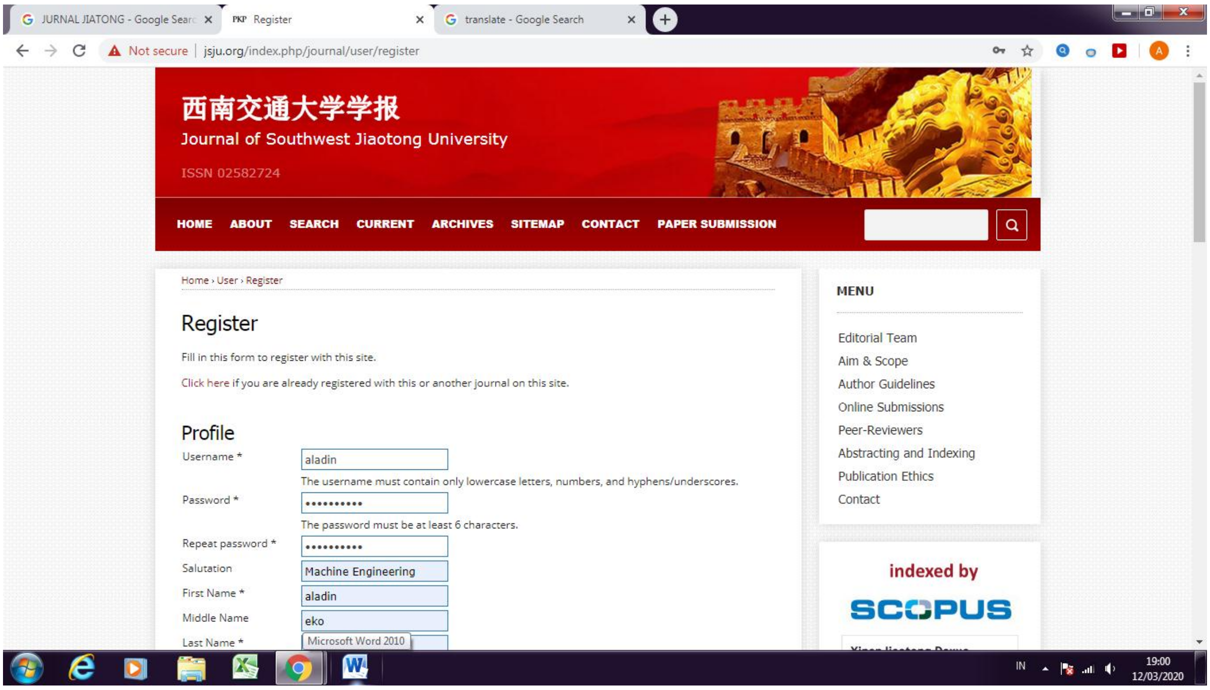

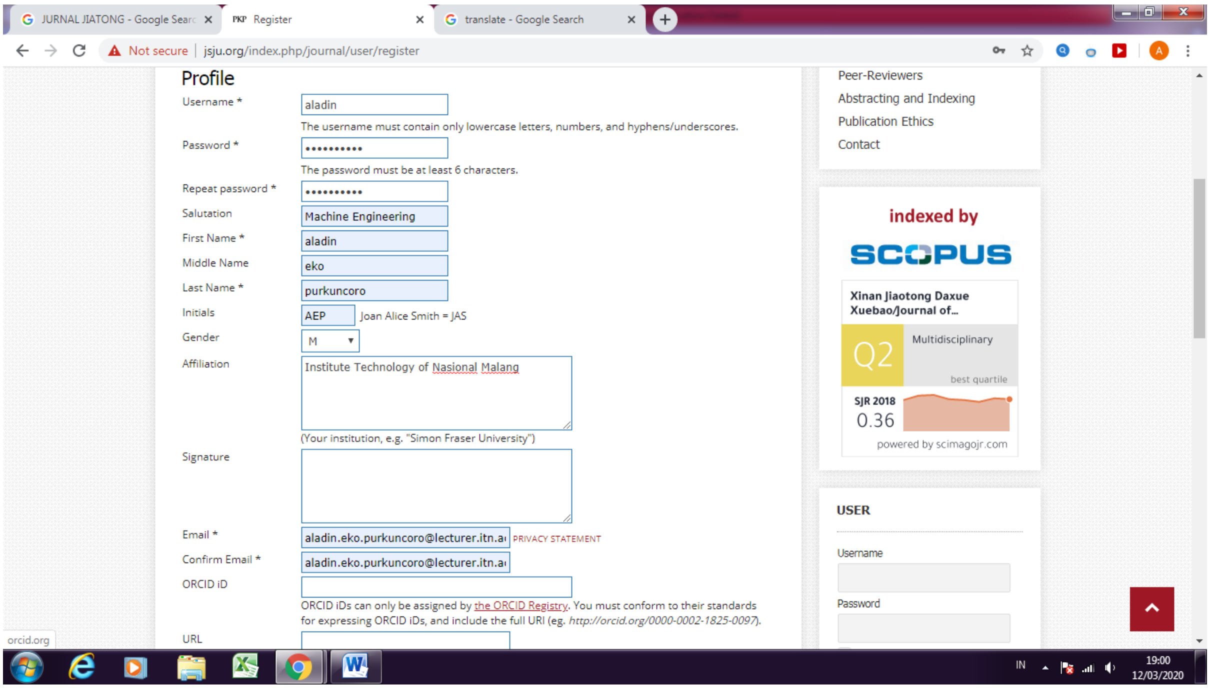

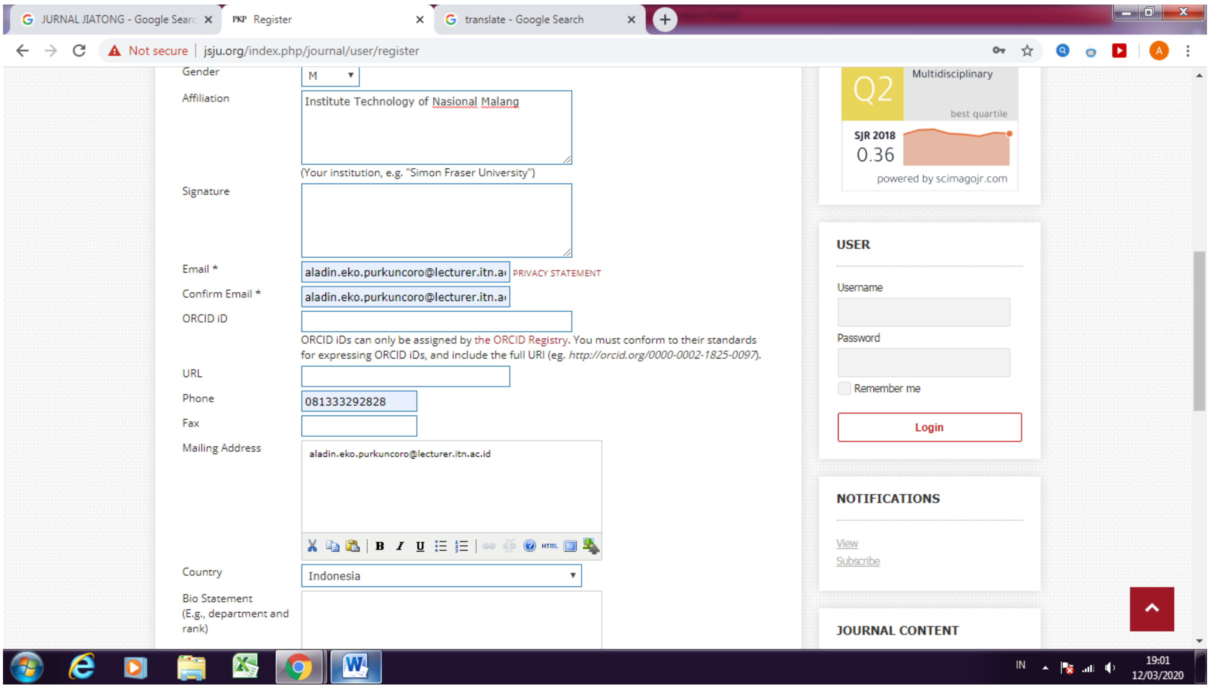
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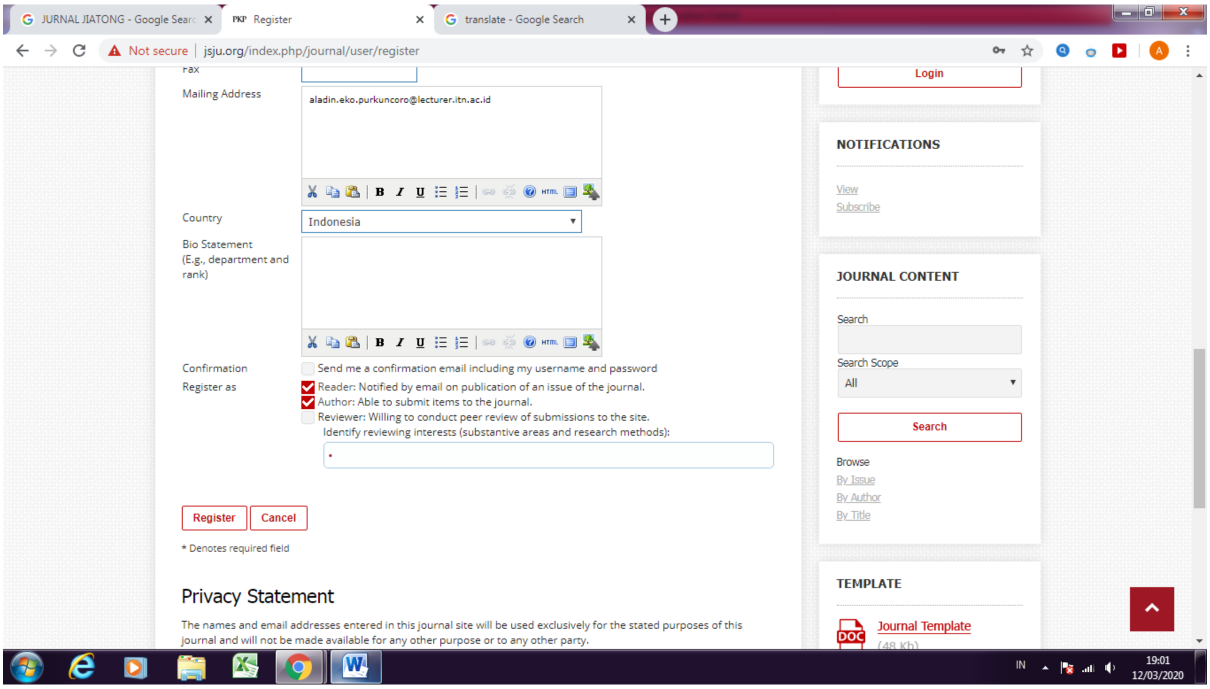
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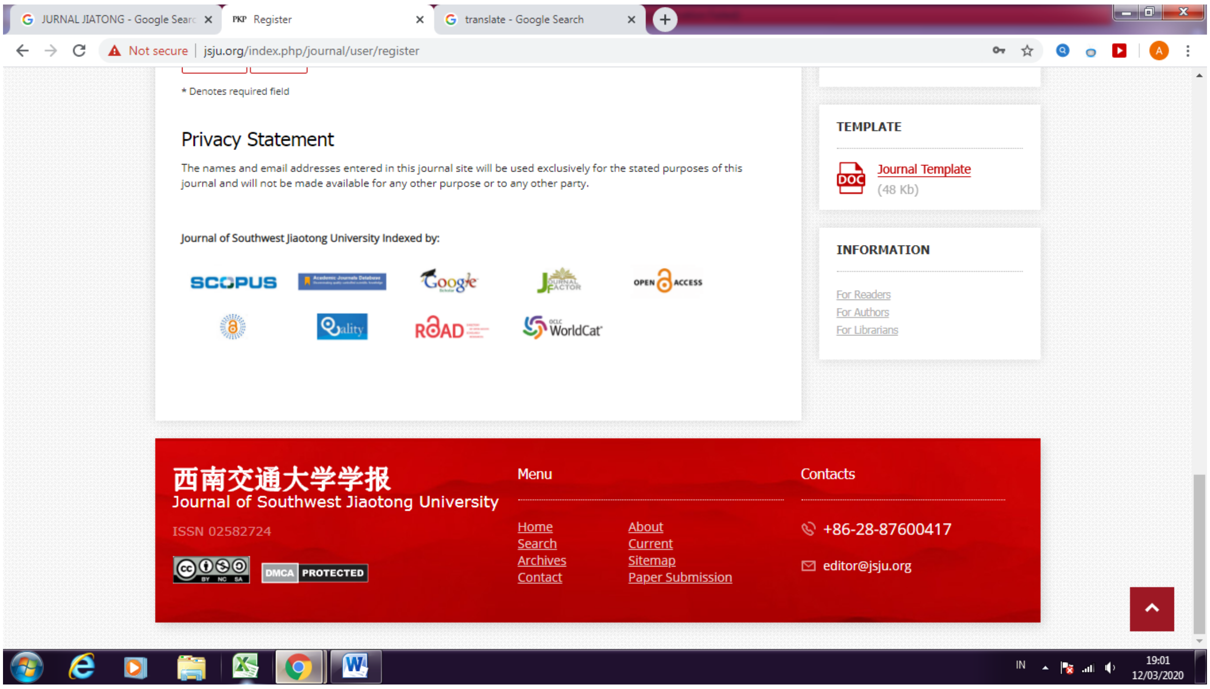
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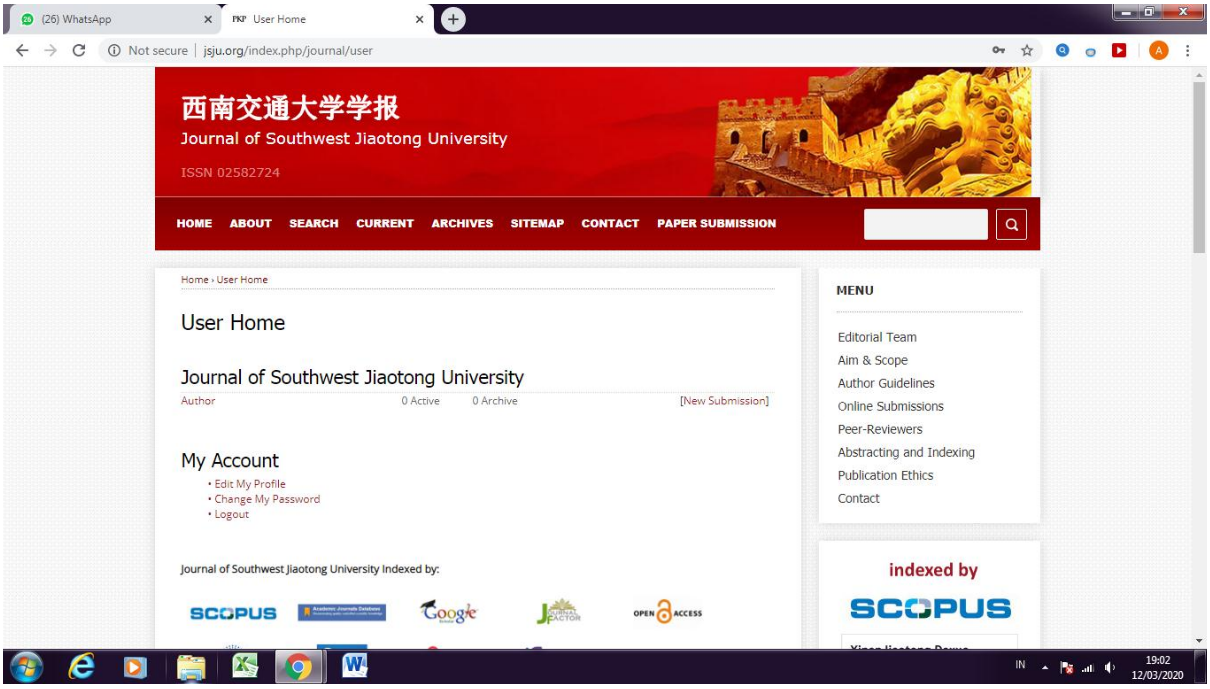
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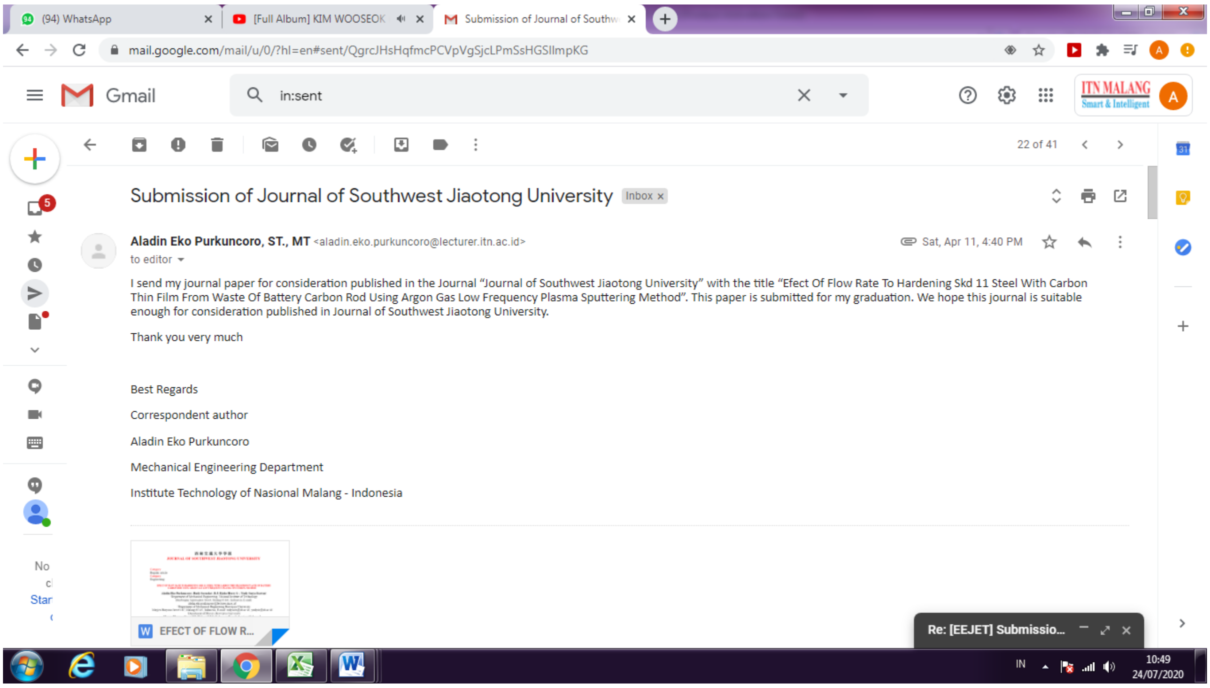

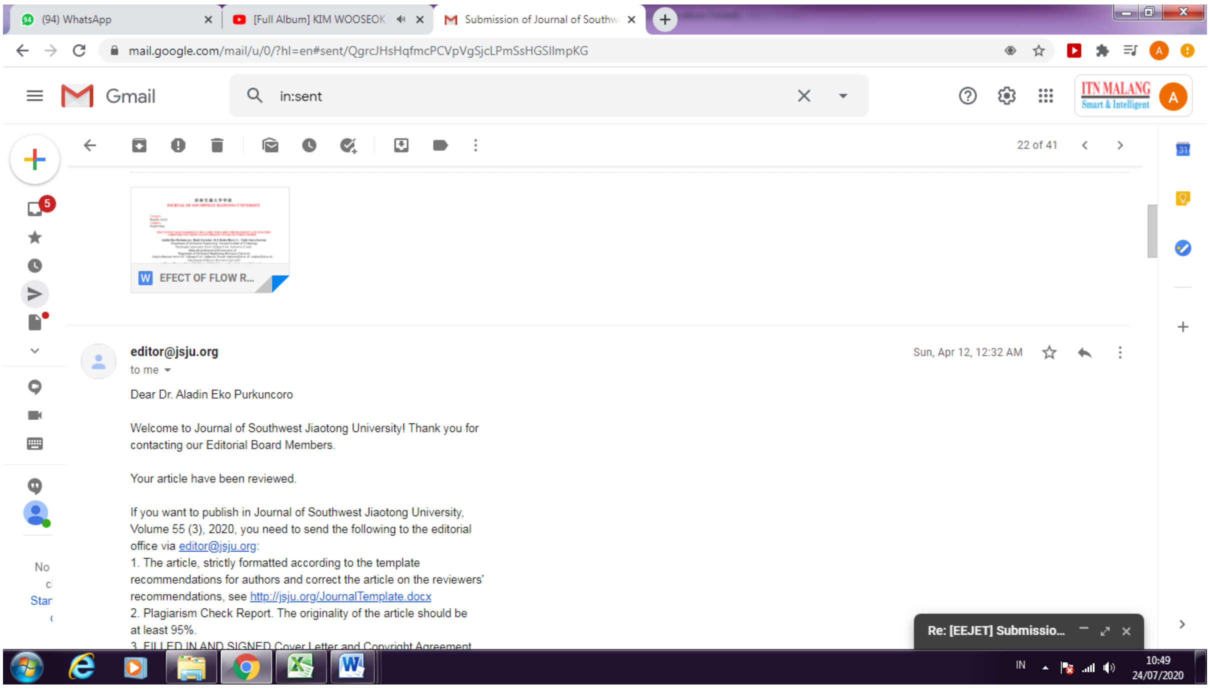

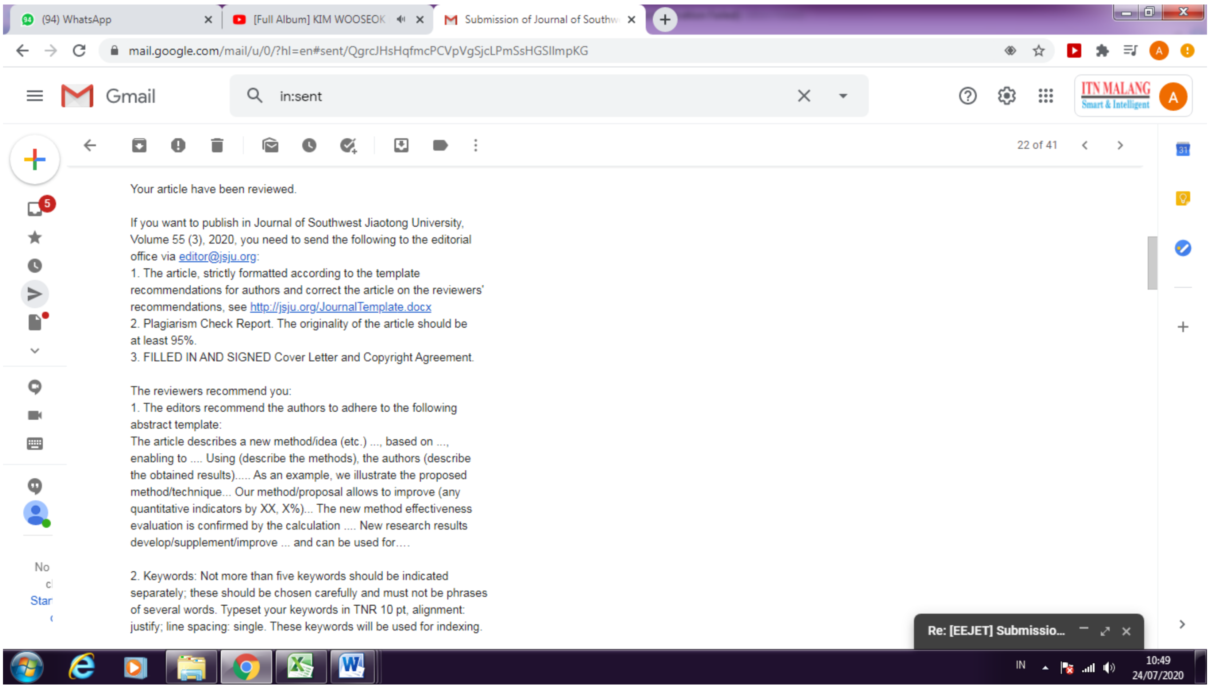

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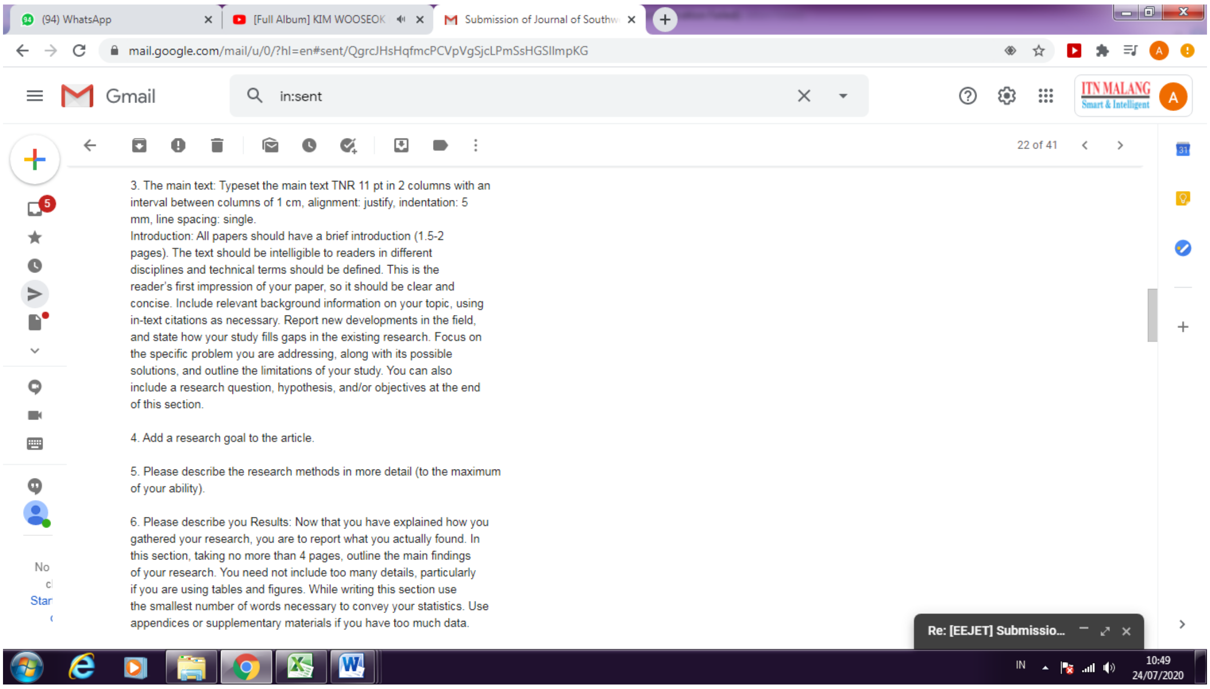

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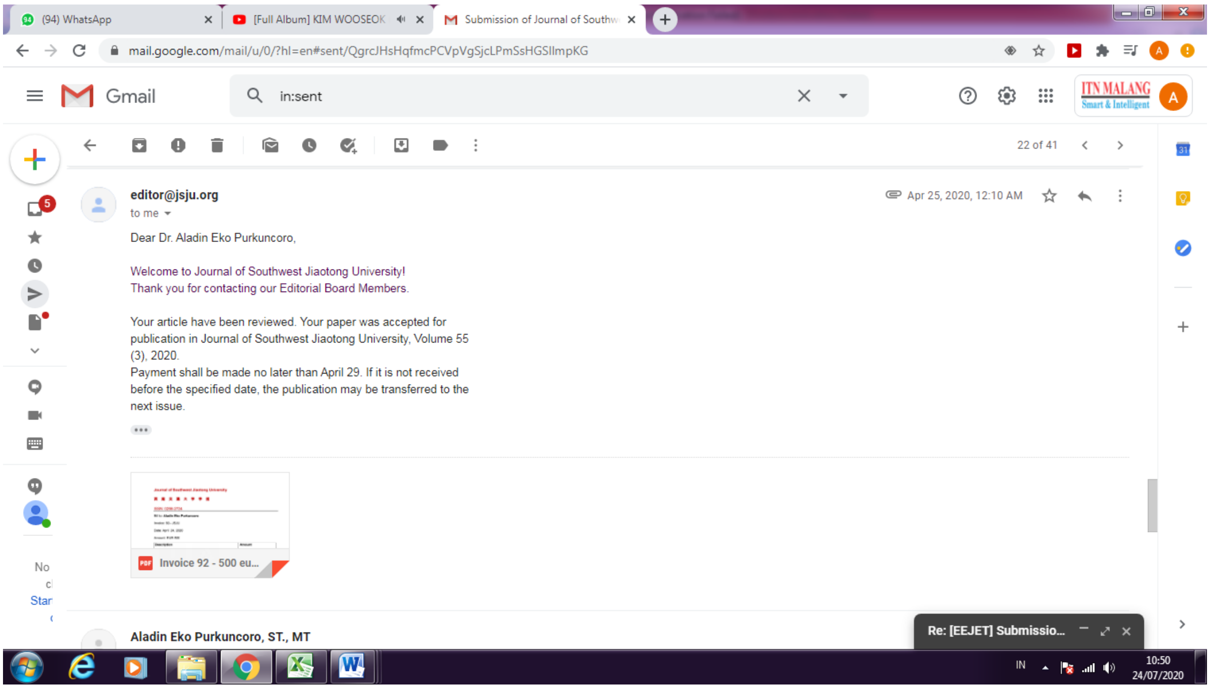

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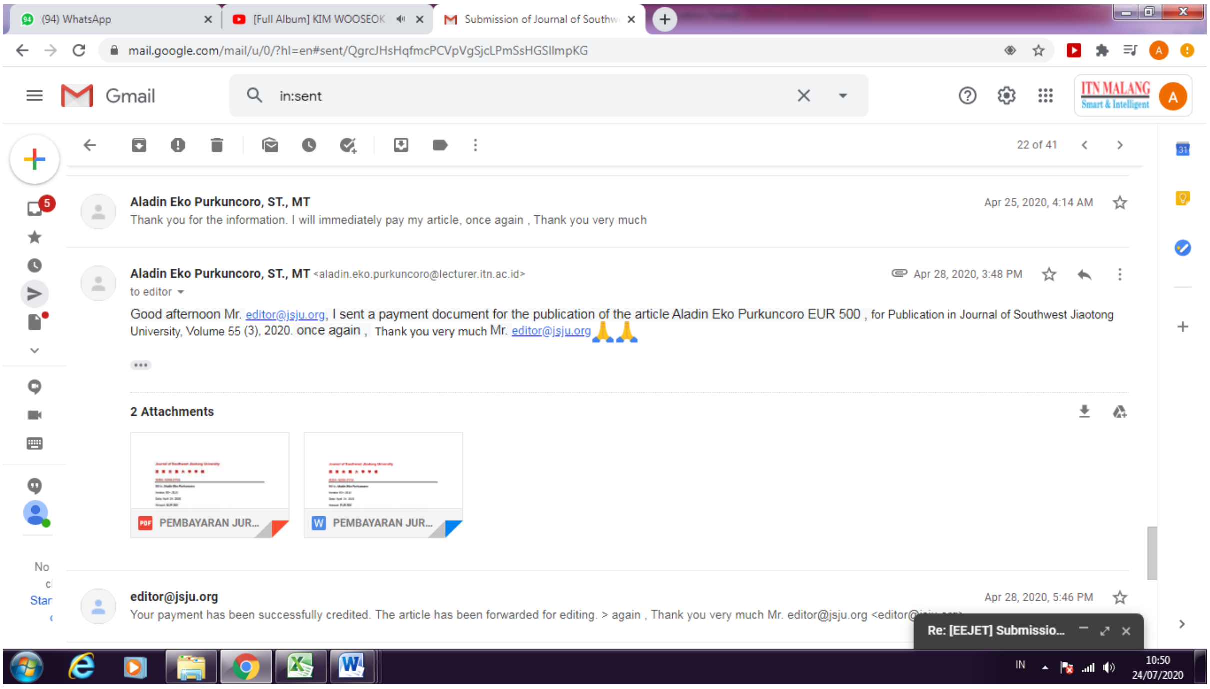

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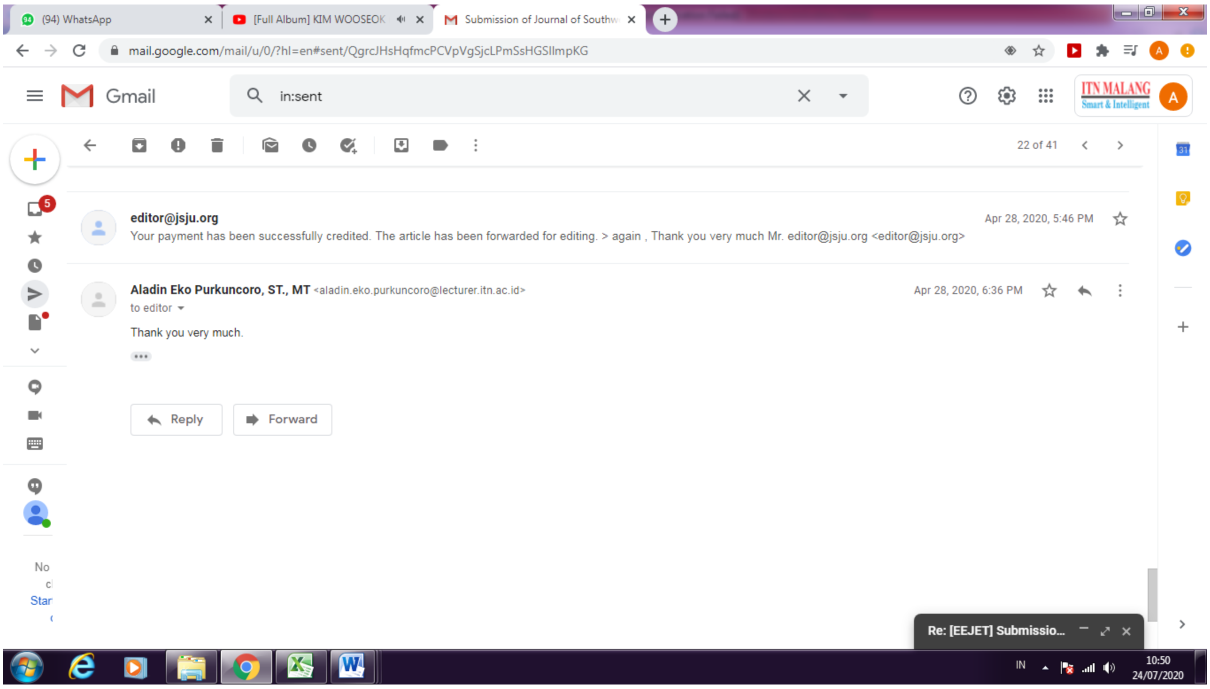
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☐ Debit Rek./*Debit Acc. No.*

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Biaya/*Charge*	Valas/*Amount in Foreign Exchange*	Kurs/ *Amount*	Nilai/ *Total Amount*
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Total yang dibayarkan/*Total Amount* :			Rp9.046.590

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Abstract

Processes for increasing the hardness value of steel are among the most challenging in industrial applications. SKD11 steel is one of the steels that is commonly used in industry (building, transportation facility construction, and production of various kinds of scissors such as knife-edge mold, scissors, circular saw blades, and metal stamping mold). However, SKD11 steel has a hardness value of only around 58–60 HV. Even though it is often used for building and transportation facility construction, which requires material with a high hardness performance. It will be better if the hardness performance is increased. The deposition of carbon thin films with plasma sputtering is a physical vapor deposition method that is effective for improving the hardness value of material. The purpose of this study is to obtain the maximum average hardness value of carbon thin films on SKD11 through the optimization of the argon gas flow rate parameter on plasma sputtering. The effect of the argon gas flow rate parameter on the hardness value of carbon thin films for SKD11 steel is investigated using a Vickers hardness tester. The research method involves the use of target material from battery waste (carbon rods) using argon gas low-frequency plasma sputtering with variations in the argon gas flow rate of 20, 40, 60, 80, and 100 mL/min for 2 hours at 300˚C. The method allows for the improvement of the hardness performance of carbon thin films because the argon gas flow rate affects the probability of carbon atom collision with particles in the chamber and the number of argon ions formed. This can cause different structures and hardness values of thin films. Based on the results, it can be concluded that the optimum argon gas flow rate parameter is 80 mL/min, which is associated with the highest average hardness value of 335.9 HV.

Keywords: SKD11, Plasma Sputtering, Battery Rod, Flow Rate, Hardness

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Lecture

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Regular article

Engineering

FLOW RATE EFFECTS ON HARDENING SKD11 STEEL WITH CARBON THIN FILM FROM BATTERY WASTE

电池废碳薄膜硬化 SKD11 钢的流动速率效应

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Abstract

Processes for increasing the hardness value of steel are among the most challenging in industrial applications. SKD11 steel is one of the steels that is commonly used in industry. However, SKD11 steel has a limited hardness performance. The deposition of carbon thin films with plasma sputtering is a physical vapor deposition method that is effective for improving the hardness value of material. The purpose of this study is to obtain the maximum average hardness value of carbon thin films on SKD11 through the optimization of the argon gas flow rate parameter on plasma sputtering. The effect of the argon gas flow rate parameter on the hardness value of carbon thin films for SKD11 steel is investigated using a Vickers hardness tester. The research method involves the use of target material from battery waste (carbon rods) using argon gas low-frequency plasma sputtering with variations in the argon gas flow rate of 20, 40, 60, 80, and 100 mL/min for 2 hours at 300°C. The method allows for the improvement of the hardness performance of carbon thin films because the argon gas flow rate affects the probability of carbon atom collision with particles in the chamber and the number of argon ions formed. This can cause different structures and hardness values of thin films. Based on the results, it can be concluded that the optimum argon gas flow rate parameter is 80 mL/min, which is associated with the highest average hardness value of 335.9 HV.

Keywords: SKD11, Plasma Sputtering, Battery Rod, Flow Rate, Hardness

摘要 提高钢的硬度值的方法是工业应用中最具挑战性的方法。SKD11 钢是工业上常用的一种钢。但是，SKD11 钢的硬度性能有限。用等离子体溅射沉积碳薄膜是一种物理气相沉积方法，对提高材料的硬度值有效。这项研究的目的是通过优化等离子溅射中的氩气流量参数来获得 SKD11 上碳薄膜的最大平均硬度值。使用维氏硬度计研究了氩气流量参数对 SKD11 钢碳薄膜硬度值的影响。

Comment [A1]: In general, it's a good idea to keep the title short so that it can be read and understood quickly. Please check this edited suggestion and make sure it's still in line with you original meaning.

Answer:
I think, the appropriate title in this research is "TH EFFECT OF ARGON FLOW RATE ON HARDNESS OF CARBON THIN FILMS ON SKD11 STEEL USING TARGET MATERIAL FROM BATTERY CARBON RODS WITH PLASMA SPUTTERING METHOD

Comment [A2]: It might be good to give an example here. What is it used for that most reade would be familiar with?

Answer:
I mean the meaning of "used in industry" is the SKD11 steel application for building, transportati facility construction, and production of various ki of scissors such as knife-edge mold, scissors, circu saw blades, and metal stamping mold.

Comment [A3]: What does this mean in concrete terms? How does the limited hardness performance affect or restrict its use in industry?

Answer:
I think, the meaning of "limited hardness performance" is not suitable, because there is sti no research that characterize the limits of hardne value of SKD11 steel.

Thus, I think the appropriate sentence is "SKD11 steel has a hardness value of only around 58–60 Even though it is often used for building and transportation facility construction, which require material with a high hardness performance. It wi be better if the hardness performance is increase

该研究方法涉及使用氩气低频等离子溅射，以 20、40、60、80 和 100 毫升/分钟的氩气流速在 20 ℃/小时变化的电池废料（碳棒）中的目标材料 2 小时。 300° C。该方法允许改善碳薄膜的硬度性能，因为氩气的流速影响碳原子与室中颗粒碰撞的可能性以及形成的氩离子的数量。这会导致薄膜的结构和硬度值不同。根据结果可以得出结论，最佳氩气流速参数为 80 毫升/分钟，这与 335.9 高压的最高平均硬度值有关。

关键词： SKD11，等离子溅射，电池棒，流量，硬度

I. Introduction

The zinc–carbon battery has been used extensively for various applications. However, the increasing use of batteries causes battery waste to increase [1], [2], and battery waste causes environmental pollution. It is important to reuse or recycle battery waste and its components.

The battery carbon rod is a battery component that contains carbon [3]. Methods for the utilization of carbon rods from zinc–carbon waste have been developed that include recycling as biogas desulfurizer [4]. Methods are also being developed to improve the hardness performance of steel [5]. SKD11 steel is one of the steels that is commonly used in the industrial world [6]. However, the steel has a limited hardness performance. SKD11 steel has a hardness value of only around 58–60 HV [7], even though it is often used for building and transportation facility construction, which requires material with a high hardness performance [6], [7], [8]. Based on this problem, the hardness performance of SKD11 steel needs to be improved. In other studies, the hardness of SKD11 steel was improved through changes in the microstructural properties of SKD11 steel during carbide dispersion carburizing. Research results show that the carbon content of the surface region in SKD11 increased by up to 3 %. The higher the carbon content of a material, the higher the hardness value. As the carbide dispersion carburizing process is generally performed at 980˚C for 120 minutes, the process requires high temperatures, consumes a lot of power, and involves a high cost [8]. Plasma sputtering is another method to increase the hardness of SKD11, but it is simpler and cheaper than carburizing [8], [9], [10].

Plasma sputtering has several advantages, such as a high deposition rate and high-quality synthesis of carbon thin films [11]. It is initiated by the bombardment of energetic ions at the target [12]. Battery waste from carbon rods can be used as a target material for the synthesis of thin films on the substrate with plasma sputtering [11]. One gas that is suitable for plasma sputtering is argon gas because argon atoms are heavy with low reactive properties and high levels of physical reaction [13]. It could be more effective than other gases for the deposition of carbon thin films [14].

In other work, the deposition of carbon thin films on glass substrate through the use low-frequency plasma sputtering with battery carbon rods as a target material has been characterized by Fourier-transform infrared (FTIR) spectroscopy. The FTIR results show C–C and C=C bonds, thus proving that carbon from battery waste (carbon rods) could be used as a target material to be successfully deposited on glass substrate [11]. In this study, carbon rods from zinc–carbon battery waste were used as a target material for the deposition of carbon thin films on SKD11 steel through argon plasma sputtering generated by a low-frequency generator at 40 kHz. The characterization of carbon thin films on SKD11 steel was carried out by using a Vickers hardness tester. The goal of this research is to obtain the maximum average hardness value of carbon thin films on SKD11 through the optimization of the argon gas flow rate parameter on plasma sputtering.

II. Material and Methods

A. Material

SKD11 steel without treatment was prepared for the experiment as substrate material. It has a 60 HV average hardness value and a chemical composition of Fe = 1.58 wt%, C = 11.4 wt%, Cr = 0.8 wt%, and Mo = 0.25 wt%. Carbon rods from zinc–carbon ABC battery waste (ABC batteries are made in Indonesia) were used as the target material in this research. The carbon purity level of the carbon rods is 72.81 wt%. Each carbon rod was cut into a diameter of 8 mm and a length of 55 mm. Then, the carbon rod was cleaned by ultrasonication in soap solution for 1 minute. The cleaned carbon rod was dried in the oven at 300˚C for 2 hours.

B. Methods

Carbon films were deposited using plasma sputtering. Figure 1a shows the details of the plasma sputtering system with a carbon rod

Comment [A4]: Once again, an example might be helpful. What are these batteries typically used for (th would be familiar to a general reader)?

Answer:
I think the appropriate sentence is "The zinc–carbon battery has been used extensively for energy source f electronic devices such as radio, camera, and remote control"

Comment [A5]: This is important, indeed. I sugges explicitly pointing out that the method described here involves reusing battery parts that would otherwise contribute to pollution.

Answer:
Yes, I agree with you and I think the appropriate sentence is "It is important to reuse waste of battery parts. One of method that can be used to reuse waste battery parts is to use carbon rods as a target materia for deposition thin films with plasma sputtering, thus will reduce pollution of battery waste"

Comment [A8]: Please check and confirm.

Answer:
I confirm that the word that should be is "Fourier-transform infrared spectroscopy (FTIR)"

Comment [A6]: Why is this bad? What would be possible with SKD11 steel if it had a higher hardness value?

Answer:
I don't think that's bad, maybe the appropriate senter is "SKD11 steel has a hardness value of only around 58 60 HV. Even though it is often used for building and transportation facility construction, which requires material with a high hardness performance. It will be better if the hardness performance is increased"

This is possible, if SKD11 steel had a higher hardness value. One of the methods that can increase the hardness value is deposition thin films on SKD11 steel with plasma sputtering method.

Comment [A9]: So that your results can be replicated by other researchers, I suggest including th full name and location of the battery manufacturer. Also, does the maker of the battery have any effect o the outcome of your experiment? Would a battery fro a different manufacturer perform differently?

Answer:
I agree with your opinion, these batteries are manufactured by PT. Everbright Manufacturing and Distribution Company at Binjai Km 9.5, 20127, Lalang Island, North Sumatra, Indonesia.

Comment [A10]: Was there only one carbon rod, did you test several carbon rods? Was only one rod u for the series of tests at different flow rates, or were different rods from different batteries used for the te at various flow rates (20, 40, 60, 80, and 100 mL/min)

I suggest outlining the specific steps of the experimen procedure a bit more specifically and clearly.

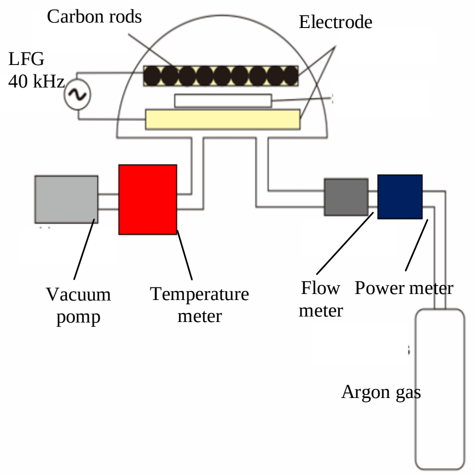
Answer:

Comment [A7]: How is this done in a laboratory?

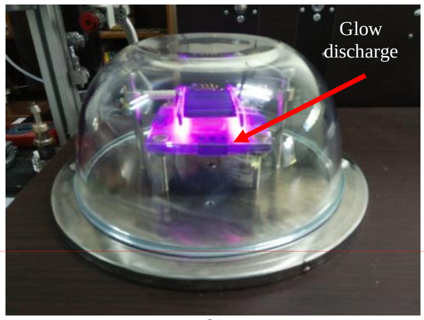
Answer:
Previous research reference were conducted in laboratory with magnetron plasma sputtering.

target. Plasma is generated by a low-frequency generator (LFG) at 40 KHz. The plasma system has a chamber diameter of 250 mm. The carbon rod as the target material was placed at the electrode, and the distance between the target material and SKD11 steel was kept at 1.7 cm. A vacuum was applied in the chamber for 1.5 hours. Argon gas then flowed into the chamber with a flow rate 20, 40, 60, 80, and 100 mL/min. The deposition was done for 2 hours, keeping the temperature at 300˚C. The procedure for the deposition of carbon thin films is developed in several steps, as described below.

1. The plasma chamber is opened and then cleaned with technical alcohol. SKD11 steel as substrate is inserted.
2. Plasma chamber is closed and vacuumed for 30 minutes with an initial temperature of 27°C
3. The temperature indicator is changed to 300°C and awaited until it reaches 300°C. Then, vacuum again for 30 minutes.
4. Flow rate of argon gas is turned on by several variations, 20, 40, 60, 80 and 100 mL/min for 30 minutes in vacuum condition.
5. The power parameter is set at 340 Watt for one hour until glow discharge appears as shown in Figure 1b and the deposition process is carried out for 2 hours.
6. The parameters of power, flow rate, temperature, vacuum pump are turned off. Then the deposited thin films on SKD11 steel is removed from plasma sputtering system.



(a)



(b)

Figure 1. (a) Schematic illustration of plasma sputtering with carbon rods-target, (b) Plasma sputtering process

C. Characterization

The content of the carbon rod and SKD11 steel was measured using SEM-EDX, Fei, Type Inspet-21**.** The hardness value of SKD11 steel, which has been deposited with carbon thin films with flow rate treatment and without said treatment, was tested using micro hardness Vickers at three different test points, as shown in Figure 2. SKD11 steel hardness is obtained by taking the average hardness value from measurements at three test points, which have been conducted previously.

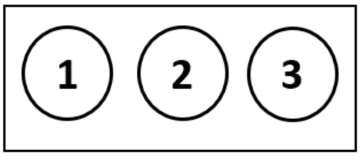


Figure 2. Point of hardness test observation

III. Results and Discussion

Red square shown in Figure 3, is the area that was observed to find out compositions of battery carbon rods, shown as Table 1.

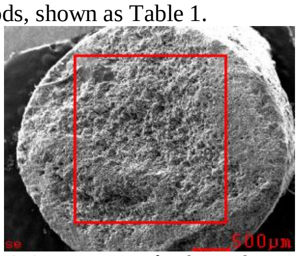


Figure 3. SEM imaging of carbon rods outer layer

Table 1.
Compositions of battery carbon rods based on SEM-EDX results

Element	Wt %
C	72.81
O	16.54
Al	1.95
Si	2.41
Cl	1.42
K	0.99

Comment [A11]: Was the gas flow rate gradually increased from 20 to 100 mL/min durin one experiment with one rod, or was there time between each flow rate with a new rod?

Answer:
There was time in between each flow rate with n rod.

Comment [A12]: How is this different from regular rubbing alcohol?

Answer:
It is same with regular rubbing alcohol, because t alcohol that used in this study consisting of rough 70% pure ethanol or isopropyl alcohol in its concentrated form.

Ca	1.29
Fe	2.59

Based on SEM-EDX results, the carbon purities level of carbon rods was relatively small (72.81 Wt %). It shows that carbon of battery carbon rods has impurities. The hardness value of SKD11 without treatment, based on the microhardness Vickers test shown in Table 2, has a relatively small hardness value.

Table 2.
The hardness value of SKD11 steel without plasma sputtering treatment based on the microhardness Vickers test

Test point	Hardness value (HV) of SKD11 steel without plasma sputtering treatment
1	60.10
2	58.20
3	59.20
Average of hardness value	59.16

The hardness value of thin films on SKD11 steel, with variation of flow rate treatment, show different hardness values at each flow rate parameter, as shown in Table 3.

Table 3.
The hardness value of thin films on SKD11 steel with flow rate treatment based on the microhardness Vickers test

Test point	Hardness value (HV) carbon thin film with argon flow rate (mL/min)				
	20	40	60	80	100
1	328.3	235.6	321.9	379.9	239.6
2	309.1	246.3	280.1	321.6	232.3
3	312.7	253.8	291.6	306.3	252.2
Average of hardness	316.7	245.2	297.9	335.9	241.3

Based on Tables 2 and 3, the average of hardness value of SKD11 steel without plasma sputtering treatment was relatively small when compared to carbon thin film with argon flow rate treatment. It indicates that the hardness value of SKD11 increases after argon flow rate on plasma sputtering treatment. Based on the results of the hardness test using micro hardness Vickers, as shown in Table 3, the treatment of flow rates with highest hardness values is obtained at a flow rate of 80 mL/ min, with average hardness values of 335.9 HV. This is caused by the probability of carbon atoms colliding with particles in the chamber and the amount of argon ions formed on plasma sputtering. The amount of argon ions has an important role in the bombardment of target material. The optimum flow rate at 80 mL/min has the highest probability of collision and the highest ion density bombarding the target material. It causes the number of carbon atoms deposited in the SKD11 to increase. As a result, the hardness value of carbon thin films increases. The line graph of hardness value of carbon thin films with variation of flow rates, as shown in Figure 3, show non-linier patterns.

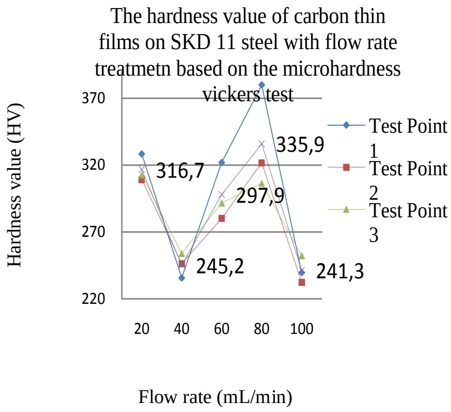


Figure 3. Line graph of hardness with flow rate

IV. Conclusions

Based on the results, it can be concluded that the optimum argon gas flow rate parameters are at 80 mL/min, having the highest average hardness of 335.9 HV. The hardness value of SKD11 steel without treatment was increased after argon flow rate on plasma sputtering treatment.

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Engineering

ARGON FLOW RATE EFFECT ON HARDNESS OF CARBON THIN FILMS ON SKD11 STEEL USING TARGET MATERIAL FROM BATTERY CARBON RODS WITH PLASMA SPUTTERING METHOD

用等离子溅射法从电池碳棒靶材上研究氩气流速对 SKD11 钢上碳薄膜硬度的影响

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Abstract

Processes for increasing the hardness value of steel are among the most challenging in industrial applications. SKD11 steel is one of the steels that is commonly used in industry (building, transportation facility construction, and production of various kinds of scissors such as knife-edge mold, scissors, circular saw blades, and metal stamping mold). However, SKD11 steel has a hardness value of only around 58–60 HV. Even though it is often used for building and transportation facility construction, which requires material with a high hardness performance. It will be better if the hardness performance is increased. The deposition of carbon thin films with plasma sputtering is a physical vapor deposition method that is effective for improving the hardness value of material. The purpose of this study is to obtain the maximum average hardness value of carbon thin films on SKD11 through the optimization of the argon gas flow rate parameter on plasma sputtering. The effect of the argon gas flow rate parameter on the hardness value of carbon thin films for SKD11 steel is investigated using a Vickers hardness tester. The research method involves the use of target material from battery waste (carbon rods) using argon gas low-frequency plasma sputtering with variations in the argon gas flow rate of 20, 40, 60, 80, and 100 mL/min for 2 hours at 300˚C. The method allows for the improvement of the hardness performance of carbon thin films because the argon gas flow rate affects the probability of carbon atom collision with particles in the chamber and the number of argon ions formed. This can cause different structures and hardness values of thin films. Based on the results, it can be concluded that the optimum argon gas flow rate parameter is 80 mL/min, which is associated with the highest average hardness value of 335.9 HV.

Keywords: SKD11, Plasma Sputtering, Battery Rod, Flow Rate, Hardness

摘要 提高钢的硬度值的方法是工业应用中最具挑战性的方法。SKD11 钢是工业（建筑，运输设施建设以及生产各种剪刀，如刀口模具，剪刀，圆锯片和金属冲压模具）中常用的一种钢。但是，SKD11 钢的硬度值仅为 58–60 高压。即使它经常用于建筑和运输设施的建设中，也需要具有高硬度性能的材料。如果提高硬度性能会更好。用等离子体溅射沉积碳薄膜是一种物理气相沉积方法，对于提高材料的硬度值是有效的。这项研究的目的是通过优化等离子溅射中的氩气流量参数来获得 SKD11 上碳薄膜的最大平均硬度值。使用维氏硬度计研究了氩气流量参数对 SKD11 钢碳薄膜硬度值的影响。该研究方法涉及使用氩气低频等离子溅射，以 20、40、60、80 和 100 毫升/分钟的氩气流速在 20℃/小时变化的电池废料（碳棒）中的目标材料 2 小时。300°摄氏度。该方法允许改善碳薄膜的硬度性能，因为氩气流速影响碳原子与腔室中的粒子碰撞的可能性以及形成的氩离子的数量。这会导致薄膜的结构和硬度值不同。根据结果可以得出结论，最佳氩气流速参数为 80 毫升/分钟，这与 335.9 高压的最高平均硬度值有关。

关键词: SKD11，等离子溅射，电池棒，流量，硬度

I. INTRODUCTION

The zinc–carbon battery has been used extensively for energy source for electronic devices such as radio, camera, and remote control. However, the increasing use of batteries causes battery waste to increase [1], [2], and battery waste causes environmental pollution. It is important to reuse waste of battery parts. One of method that can be used to reuse waste of battery parts is to use carbon rods as a target material for deposition thin films with plasma sputtering, thus it will reduce pollution of battery waste.

The battery carbon rod is a battery component that contains carbon [3]. Methods for the utilization of carbon rods from zinc–carbon waste have been developed that include recycling as biogas desulfurizer [4]. Methods are also being developed to improve the hardness performance of steel [5]. SKD11 steel is one of the steels that is commonly used in the industrial world [6]. However, the steel has a limited hardness performance. SKD11 steel has a hardness value of only around 58–60 HV [7], even though it is often used for building and transportation facility construction, which requires material with a high hardness performance. It will be better if the hardness performance is increased [6], [7], [8]. This is possible, if SKD11 steel had a higher hardness value. One of the methods that can increase the hardness value is deposition thin films on SKD11 steel with plasma sputtering method. Based on this problem, the hardness performance of SKD11 steel needs to be improved. In other studies, the hardness of SKD11 steel was improved through changes in the microstructural properties of SKD11 steel during carbide dispersion carburizing. Research results show that the carbon content of the surface region in SKD11 increased by up to 3 %. The higher the carbon content of a material, the higher the hardness value. As the carbide dispersion carburizing process is generally performed at 980˚C for 120 minutes, the process requires high temperatures, consumes a lot of power, and involves a high cost [8]. Plasma sputtering is another method to increase the hardness of SKD11, but it is simpler and cheaper than carburizing [8], [9], [10].

Plasma sputtering has several advantages, such as a high deposition rate and high-quality synthesis of carbon thin films [11]. It is initiated by the bombardment of energetic ions at the target [12] (Previous research reference were conducted in laboratory with magnetron plasma sputtering.). Battery waste from carbon rods can be used as a target material for the synthesis of thin films on the substrate with plasma sputtering [11]. One gas that is suitable for plasma sputtering is argon gas because argon atoms are heavy with low reactive properties and high levels of physical reaction [13]. It could be more effective than other gases for the deposition of carbon thin films [14].

In other work, the deposition of carbon thin films on glass substrate through the use low-frequency plasma sputtering with battery carbon rods as a target material has been characterized by Fourier-transform infrared spectroscopy (FTIR). The FTIR results show C–C and C=C bonds, thus proving that carbon from battery waste (carbon rods) could be used as a target material to be successfully deposited on glass substrate [11]. In this study, carbon rods from zinc–carbon battery waste were used as a target material for the deposition of carbon thin films on SKD11 steel through argon plasma sputtering generated by a low-frequency generator at 40 kHz. The characterization of carbon thin films on SKD11 steel was carried out by using a Vickers hardness tester. The goal of this research is to obtain the maximum average hardness value of carbon thin films on SKD11 through the optimization of the argon gas flow rate parameter on plasma sputtering.

II. Material and Methods

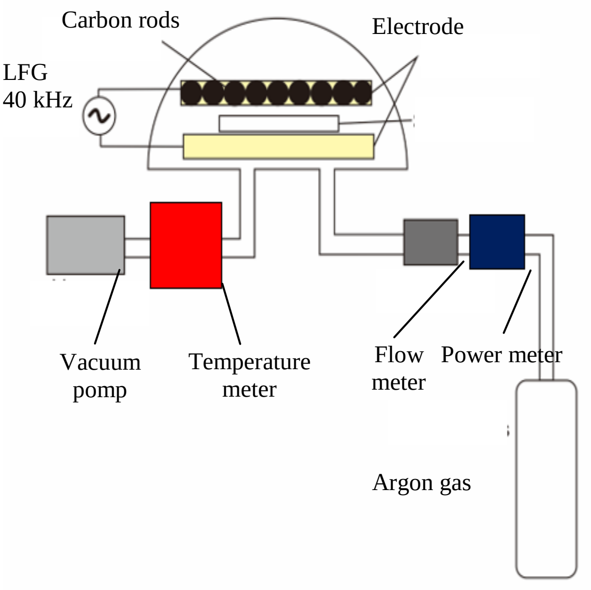
A. Material

SKD11 steel without treatment was prepared for the experiment as substrate material. It has a 60 HV average hardness value and a chemical composition of Fe = 1.58 wt%, C = 11.4 wt%, Cr = 0.8 wt%, and Mo = 0.25 wt%. Carbon rods from zinc–carbon ABC battery waste (ABC batteries are manufactured by PT. Everbright Manufacturing and Distribution Company at Binjai Km 9.5, 20127, Lalang Island, North Sumatra, Indonesia. The maker of the battery has an effect on the outcome of the experiment if the chemical composition of the battery is different with the maker of the battery. Different manufacturer perform differently of the chemical composition of the battery is different.) were used as the target material in this research. The experiment was carried out using 9 carbon rods in plasma sputtering system from the same batteries that manufactured by the above-mentioned company for test at various flow rates. The carbon purity level of the carbon rods is 72.81 wt%. Each carbon rod was cut into a diameter of 8 mm and a length of 55 mm. Then, the carbon rod was cleaned by ultrasonication in soap solution for 1 minute. The cleaned carbon rod was dried in the oven at 300˚C for 2 hours.

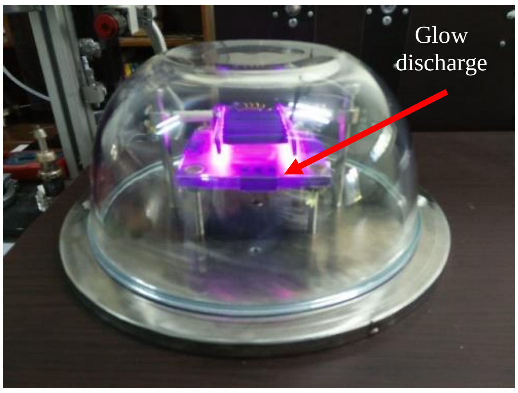
B. Methods

Carbon films were deposited using plasma sputtering. Figure 1a shows the details of the plasma sputtering system with a carbon rod target. Plasma is generated by a low-frequency generator (LFG) at 40 KHz. The plasma system has a chamber diameter of 250 mm. The carbon rod as the target material was placed at the electrode, and the distance between the target material and SKD11 steel was kept at 1.7 cm. A vacuum was applied in the chamber for 1.5 hours. Argon gas then flowed into the chamber with a flow rate 20, 40, 60, 80, and 100 mL/min. There was time in between each flow rate with new rod. The deposition was done for 2 hours, keeping the temperature at 300˚C. The procedure for the deposition of carbon thin films is developed in several steps, as described below.

1. The plasma chamber is opened and then cleaned with technical alcohol (the same with regular rubbing alcohol, because the alcohol that used in this study consisting of roughly 70% pure ethanol or isopropyl alcohol in its concentrated form). SKD11 steel as substrate is inserted.
2. Plasma chamber is closed and vacuumed for 30 minutes with an initial temperature of 27°C
3. The temperature indicator is changed to 300°C and awaited until it reaches 300°C. Then, vacuum again for 30 minutes.
4. Flow rate of argon gas is turned on by several variations, 20, 40, 60, 80 and 100 mL/min for 30 minutes in vacuum condition.
5. The power parameter is set at 340 Watt for one hour until glow discharge appears as shown in Figure 1b and the deposition process is carried out for 2 hours.
6. The parameters of power, flow rate, temperature, vacuum pump are turned off. Then the deposited thin films on SKD11 steel is removed from plasma sputtering system.



(a)



(b)

Figure 1. (a) Schematic illustration of plasma sputtering with carbon rods-target, (b) Plasma sputtering process

C. Characterization

The content of the carbon rod and SKD11 steel was measured using SEM-EDX, Fei, Type Inspet-21**.** The hardness value of SKD11 steel, which has been deposited with carbon thin films with flow rate treatment and without said treatment, was tested using micro hardness Vickers at three different test points, as shown in Figure 2. SKD11 steel hardness is obtained by taking the average hardness value from measurements at three test points, which have been conducted previously.

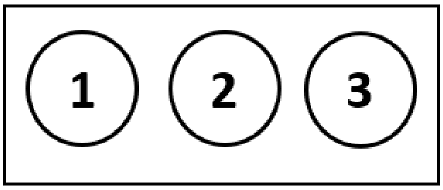


Figure 2. Point of hardness test observation

III. Results and Discussion

Red square shown in Figure 3, is the area that was observed to find out compositions of battery carbon rods, shown as Table 1.

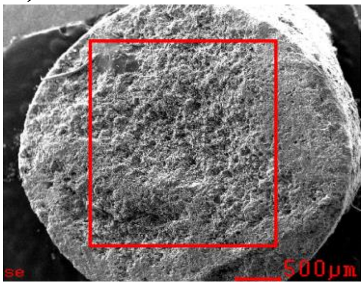


Figure 3. SEM imaging of carbon rods outer layer

Table 1.
Compositions of battery carbon rods based on SEM-EDX results

Element	Wt %
C	72.81
O	16.54
Al	1.95
Si	2.41
Cl	1.42
K	0.99
Ca	1.29
Fe	2.59

Based on SEM-EDX results, the carbon purities level of carbon rods was relatively small (72.81 Wt %). It shows that carbon of battery carbon rods has impurities. The hardness value of SKD11 without treatment, based on the microhardness Vickers test shown in Table 2, has a relatively small hardness value.

Table 2.
The hardness value of SKD11 steel without plasma sputtering treatment based on the microhardness Vickers test

Test point	Hardness value (HV) of SKD11 steel without plasma sputtering treatment
1	60.10
2	58.20
3	59.20
Average of hardness value	59.16

The hardness value of thin films on SKD11 steel, with variation of flow rate treatment, show different hardness values at each flow rate parameter, as shown in Table 3.

Table 3.
The hardness value of thin films on SKD11 steel with flow rate treatment based on the microhardness Vickers test

Test point	Hardness value (HV) carbon thin film with argon flow rate (mL/min)				
	20	40	60	80	100
1	328.3	235.6	321.9	379.9	239.6
2	309.1	246.3	280.1	321.6	232.3
3	312.7	253.8	291.6	306.3	252.2
Average of hardness	316.7	245.2	297.9	335.9	241.3

Based on Tables 2 and 3, the average of hardness value of SKD11 steel without plasma sputtering treatment was relatively small when compared to carbon thin film with argon flow rate treatment. It indicates that the hardness value of SKD11 increases after argon flow rate on plasma sputtering treatment. Based on the results of the hardness test using micro hardness Vickers, as shown in Table 3, the treatment of flow rates with highest hardness values is obtained at a flow rate of 80 mL/ min, with average hardness values of 335.9 HV. This is caused by the probability of carbon atoms colliding with particles in the chamber and the amount of argon ions formed on plasma sputtering. The amount of argon ions has an important role in the bombardment of target material. The optimum flow rate at 80 mL/min has the highest probability of collision and the highest ion density bombarding the target material. It causes the number of carbon atoms deposited in the SKD11 to increase. As a result, the hardness value of carbon thin films increases. The line graph of hardness value of carbon thin films with variation of flow rates, as shown in Figure 3, show non-linier patterns.

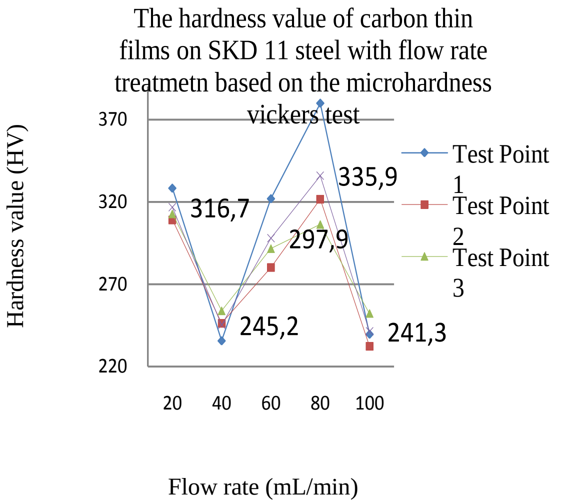


Figure 3. Line graph of hardness with flow rate

IV. Conclusions

Based on the results, it can be concluded that the optimum argon gas flow rate parameters are at 80 mL/min, having the highest average hardness of 335.9 HV. The hardness value of SKD11 steel without treatment was increased after argon flow rate on plasma sputtering treatment.

Acknowledgements

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ARGON FLOW RATE EFFECT ON HARDNESS OF CARBON THIN FILMS ON SKD11 STEEL USING TARGET MATERIAL FROM BATTERY CARBON RODS WITH PLASMA SPUTTERING METHOD

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Engineering

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用等离子溅射法从电池碳棒靶材上研究氩气流速对 SKD11 钢上碳薄膜硬度的影响

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Abstract

Processes for increasing the hardness value of steel are among the most challenging in industrial applications. SKD11 steel is one of the steels that is commonly used in industry (building, transportation facility construction, and production of various kinds of scissors such as knife-edge mold, scissors, circular saw blades, and metal stamping mold). However, SKD11 steel has a hardness value of only around 58–60 HV. Even though it is often used for building and transportation facility construction, which requires material with a high hardness performance. It will be better if the hardness performance is increased. The deposition of carbon thin films with plasma sputtering is a physical vapor deposition method that is effective for improving the hardness value of material. The purpose of this study is to obtain the maximum average hardness value of carbon thin films on SKD11 through the optimization of the argon gas flow rate parameter on plasma sputtering. The effect of the argon gas flow rate parameter on the hardness value of carbon thin films for SKD11 steel is investigated using a Vickers hardness tester. The research method involves the use of target material from battery waste (carbon rods) using argon gas low-frequency plasma sputtering with variations in the argon gas flow rate of 20, 40, 60, 80, and 100 mL/min for 2 hours at 300°C. The method allows for the improvement of the hardness performance of carbon thin films because the argon gas flow rate affects the probability of carbon atom collision with particles in the chamber and the number of argon ions formed. This can cause different structures and hardness values of thin films. Based on the results, it can be concluded that the optimum argon gas flow rate parameter is 80 mL/min, which is associated with the highest average hardness value of 335.9 HV.

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关键词: SKD11，等离子溅射，电池棒，流量，硬度

I. Introduction

The zinc–carbon battery has been used extensively for energy source for electronic devices such as radio, camera, and remote control. However, the increasing use of batteries causes battery waste to increase [1], [2], and battery waste causes environmental pollution. It is important to reuse waste of battery parts. One of method that can be used to reuse waste of battery parts is to use carbon rods as a target material for deposition thin films with plasma sputtering, thus it will reduce pollution of battery waste.

The battery carbon rod is a battery component that contains carbon [3]. Methods for the utilization of carbon rods from zinc–carbon waste have been developed that include recycling as biogas desulfurizer [4]. Methods are also being developed to improve the hardness performance of steel [5]. SKD11 steel is one of the steels that is commonly used in the industrial world [6]. However, the steel has a limited hardness performance. SKD11 steel has a hardness value of only around 58–60 HV [7], even though it is often used for building and transportation facility construction, which requires material with a high hardness performance. It will be better if the hardness performance is increased [6], [7], [8]. This is possible, if SKD11 steel had a higher hardness value. One of the methods that can increase the hardness value is deposition thin films on SKD11 steel with plasma sputtering method. Based on this problem, the hardness performance of SKD11 steel needs to be improved. In other studies, the hardness of SKD11 steel was improved through changes in the microstructural properties of SKD11 steel during carbide dispersion carburizing. Research results show that the carbon content of the surface region in SKD11 increased by up to 3 %. The higher the carbon content of a material, the higher the hardness value. As the carbide dispersion carburizing process is generally performed at 980°C for 120 minutes, the process requires high temperatures, consumes a lot of power, and involves a high cost [8]. Plasma sputtering is another method to increase the hardness of SKD11, but it is simpler and cheaper than carburizing [8], [9], [10].

Plasma sputtering has several advantages, such as a high deposition rate and high-quality synthesis of carbon thin films [11]. It is initiated by the bombardment of energetic ions at the target [12] (Previous research reference were conducted in laboratory with magnetron plasma sputtering.). Battery waste from carbon rods can be used as a target material for the synthesis of thin films on the substrate with plasma sputtering [11]. One gas that is suitable for plasma sputtering is argon gas because argon atoms are heavy with low reactive properties and high levels of physical reaction [13]. It could be more effective than other gases for the deposition of carbon thin films [14].

In other work, the deposition of carbon thin films on glass substrate through the use low-frequency plasma sputtering with battery carbon rods as a target material has been characterized by Fourier-transform infrared spectroscopy (FTIR). The FTIR results show C–C and C=C bonds, thus proving that carbon from battery waste (carbon rods) could be used as a target material to be successfully deposited on glass substrate [11]. In this study, carbon rods from zinc–carbon battery waste were used as a target material for the deposition of carbon thin films on SKD11 steel through argon plasma sputtering generated by a low-frequency generator at 40 kHz. The characterization of carbon thin films on SKD11 steel was carried out by using a Vickers hardness tester. The goal of this research is to obtain the maximum average hardness value of carbon thin films on SKD11 through the optimization of the argon gas flow rate parameter on plasma sputtering.

II. Material and Methods

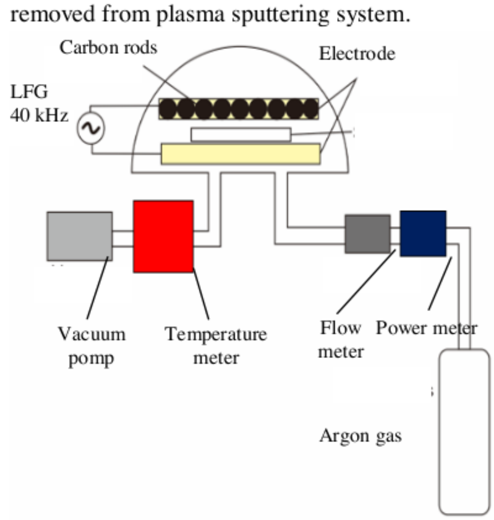
A. Material

SKD11 steel without treatment was prepared for the experiment as substrate material. It has a 60 HV average hardness value and a chemical composition of Fe = 1.58 wt%, C = 11.4 wt%, Cr = 0.8 wt%, and Mo = 0.25 wt%. Carbon rods from zinc–carbon ABC battery waste (ABC batteries are manufactured by PT. Everbright Manufacturing and Distribution Company at Binjai Km 9.5, 20127, Lalang Island, North Sumatra, Indonesia. The maker of the battery has an effect on the outcome of the experiment if the chemical composition of the battery is different with the maker of the battery. Different manufacturer perform differently of the chemical composition of the battery is different.) were used as the target material in this research. The experiment was carried out using 9 carbon rods in plasma sputtering system from the same batteries that manufactured by the above-mentioned company for test at various flow rates. The carbon purity level of the carbon rods is 72.81 wt%. Each carbon rod was cut into a diameter of 8 mm and a length of 55 mm. Then, the carbon rod was cleaned by ultrasonication in soap solution for 1 minute. The cleaned carbon rod was dried in the oven at 300˚C for 2 hours.

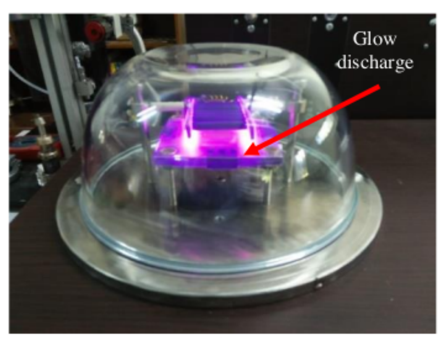
B. Methods

Carbon films were deposited using plasma sputtering. Figure 1a shows the details of the plasma sputtering system with a carbon rod target. Plasma is generated by a low-frequency generator (LFG) at 40 KHz. The plasma system has a chamber diameter of 250 mm. The carbon rod as the target material was placed at the electrode, and the distance between the target material and SKD11 steel was kept at 1.7 cm. A vacuum was applied in the chamber for 1.5 hours. Argon gas then flowed into the chamber with a flow rate 20, 40, 60, 80, and 100 mL/min. There was time in between each flow rate with new rod. The deposition was done for 2 hours, keeping the temperature at 300˚C. The procedure for the deposition of carbon thin films is developed in several steps, as described below.

1. The plasma chamber is opened and then cleaned with technical alcohol (the same with regular rubbing alcohol, because the alcohol that used in this study consisting of roughly 70% pure ethanol or isopropyl alcohol in its concentrated form). SKD11 steel as substrate is inserted.
2. Plasma chamber is closed and vacuumed for 30 minutes with an initial temperature of 27ºC
3. The temperature indicator is changed to 300ºC and awaited until it reaches 300ºC. Then, vacuum again for 30 minutes.
4. Flow rate of argon gas is turned on by several variations, 20, 40, 60, 80 and 100 mL/min for 30 minutes in vacuum condition.
5. The power parameter is set at 340 Watt for one hour until glow discharge appears as shown in Figure 1b and the deposition process is carried out for 2 hours.
6. The parameters of power, flow rate, temperature, vacuum pump are turned off. Then the deposited thin films on SKD11 steel is removed from plasma sputtering system.



(a)



(b)

Figure 1. (a) Schematic illustration of plasma sputtering with carbon rods-target, (b) Plasma sputtering process

C. Characterization

The content of the carbon rod and SKD11 steel was measured using SEM-EDX, Fei, Type Inspet-21. The hardness value of SKD11 steel, which has been deposited with carbon thin films with flow rate treatment and without said treatment, was tested using micro hardness Vickers at three different test points, as shown in Figure 2. SKD11 steel hardness is obtained by taking the average hardness value from measurements at three test points, which have been conducted previously.

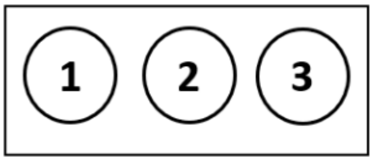


Figure 2. Point of hardness test observation

III. Results and Discussion

Red square shown in Figure 3, is the area that was observed to find out compositions of battery carbon rods, shown as Table 1.

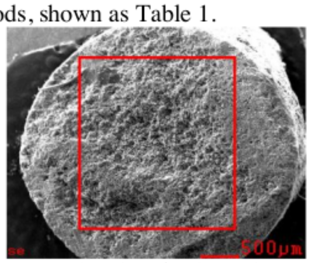


Figure 3. SEM imaging of carbon rods outer layer

Table 1.
Compositions of battery carbon rods based on SEM-EDX results

Element	Wt %
C	72.81
O	16.54
Al	1.95
Si	2.41
Cl	1.42
K	0.99
Ca	1.29
Fe	2.59

Based on SEM-EDX results, the carbon purities level of carbon rods was relatively small (72.81 Wt %). It shows that carbon of battery carbon rods has impurities. The hardness value of SKD11 without treatment, based on the microhardness Vickers test shown in Table 2, has a relatively small hardness value.

Table 2.
The hardness value of SKD11 steel without plasma sputtering treatment based on the microhardness Vickers test

Test point	Hardness value (HV) of SKD11 steel without plasma sputtering treatment
1	60.10
2	58.20
3	59.20
Average of hardness value	59.16

The hardness value of thin films on SKD11 steel, with variation of flow rate treatment, show different hardness values at each flow rate parameter, as shown in Table 3.

Table 3.
The hardness value of thin films on SKD11 steel with flow rate treatment based on the microhardness Vickers test

Test point	Hardness value (HV) carbon thin film with argon flow rate (mL/min)				
	20	40	60	80	100
1	328.3	235.6	321.9	379.9	239.6
2	309.1	246.3	280.1	321.6	232.3
3	312.7	253.8	291.6	306.3	252.2
Average of hardness	316.7	245.2	297.9	335.9	241.3

Based on Tables 2 and 3, the average of hardness value of SKD11 steel without plasma sputtering treatment was relatively small when compared to carbon thin film with argon flow rate treatment. It indicates that the hardness value of SKD11 increases after argon flow rate on plasma sputtering treatment. Based on the results of the hardness test using micro hardness Vickers, as shown in Table 3, the treatment of flow rates with highest hardness values is obtained at a flow rate of 80 mL/ min, with average hardness values of 335.9 HV. This is caused by the probability of carbon atoms colliding with particles in the chamber and the amount of argon ions formed on plasma sputtering. The amount of argon ions has an important role in the bombardment of target material. The optimum flow rate at 80 mL/min has the highest probability of collision and the highest ion density bombarding the target material. It causes the number of carbon atoms deposited in the SKD11 to increase. As a result, the hardness value of carbon thin films increases. The line graph of hardness value of carbon thin films with variation of flow rates, as shown in Figure 3, show non-linier patterns.

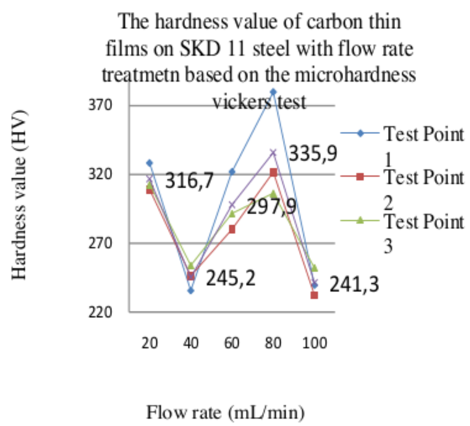


Figure 3. Line graph of hardness with flow rate

IV. Conclusions

Based on the results, it can be concluded that the optimum argon gas flow rate parameters are at 80 mL/min, having the highest average hardness of 335.9 HV. The hardness value of SKD11 steel without treatment was increased after argon flow rate on plasma sputtering treatment.

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ARGON FLOW RATE EFFECT ON HARDNESS OF CARBON THIN FILMS ON SKD11 STEEL USING TARGET MATERIAL FROM BATTERY CARBON RODS WITH PLASMA SPUTTERING METHOD

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