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Laboratory Research on Design of Three-Phase AC Arc Plasma Pyrolysis Device for Recycling of Waste Printed Circuit Boards

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Abstract: Accumulation of electronic waste (e-waste) will place a heavy burden on the environment without proper treatment; however, most ingredients contained in it are useful, and it could bring great economic benefits when recycled. A three-phase alternating current (AC) arc plasma pyrolysis device was designed for resourcing treatment of waste printed circuit boards (WPCBs). This paper focuses on the analysis of plasma pyrolysis gas products, and the results showed that the plasma could operate stably, and overcame the problems of the poor continuity and low energy of single-arc discharge. Air-plasma would generate NO_x contaminants, burn the organics, and oxidize the metals; therefore, air had not been selected as a working gas. Ar-plasma can break the long chains of organic macromolecules to make a combustible gas. Moreover, the strong adhesion between the metals and fiberglass boards would be destroyed, which facilitates subsequent separation. Ar/H_2-plasma promoted the decrease of carbon dioxide and the increase of combustible small molecular hydrocarbons in the pyrolysis product compared with Ar-plasma, and the increase of the H_2 flow rate or plasma power intensified that promotion effect. The percentage of other components, except the hydrogen of CO_2, CO, CH_4, C_2H_4, and C_3H_6, accounted for 55.7%, 34.2%, 5.6%, 4.5%, and 0% in Ar-plasma, and changed to 35.0%, 29.0%, 11.2%, 24.3%, and 0.5% in Ar/H_2-plasma. Ar/H_2-plasma could provide a highly chemically active species and break chemical bonds in organic macromolecules to produce small molecules of combustible gas. This laboratory work presents a novel three-phase AC arc plasma device and a new way for recycling WPCBs with high value.

Keywords: three-phase AC arc plasma; waste printed circuit boards; resourcing treatment

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1. Introduction

The rapid development of information science and technology brings great convenience to people's lives. At the same time, the increasing growth and accumulation of e-waste also places a heavy burden upon the environment, which has already received a great deal of attention [1]. Globally, 44.7 million tons of e-waste was produced in 2016, which grew to 49.80 million tons in 2018, and it is estimated that the number will reach 52.2 million tons by 2021, with a growth rate of 3% to 4% per year [2–4].

As the most typical electronic waste, WPCBs usually contain different heavy metals and halogenated flame retardants, which will harm the environment and human beings without proper treatment [5–7]. Furthermore, ingredients contained in WPCBs are useful, and it will also cause a great waste of resources without proper treatment. Research has found that 1 ton of WPCBs contains about 130 kg of copper (Cu), about 3.5 kg of silver (Ag), about 340 g of gold (Au), and about 140 g of palladium (Pd), the grade of which is several times the original ore grade [8–10].

Therefore, recycling WPCBs has become an increasingly debated issue in the last two decades, both in scientific research and at a government level [11]; however, the separation and recovery of WPCBs are serious environmental challenges, because this process is usually accompanied by the generation of harmful substances [12–14]. Up to now, several methods have been developed to dispose of WPCBs and recycle useful metals, such as smelting, leaching, solvent extraction, and electrochemistry [15]. Mechanical and physical methods are currently preferred to separate and recover different metals from WPCBs, as they add a high degree of value, they are low cost, and they cause no environmental pollution [16–19]. The integrated method includes two main processes: liberation by crushing and separation by physical technology. The physical separation technologies include magnetic separation, eddy current separation, corona electrostatic separation, and so on [15]. Xue et al. successfully recovered Cu, Fe and Al and plastics from WPCBs using physical and mechanical methods, and the purity of Cu exceeded 98% [20,21]. Hydrometallurgy involving chemical leaching is also widely used because it is highly effective for metal recovery [22–24]. In general, the hydrometallurgy method also involves two steps, including mechanical crushing and acidic leaching. This method requires the use of large amounts of acid, resulting in a high cost and adverse effects on the ecosystem [25,26]. The application of microbial activities in metal recycling is also increasingly explored as a green technology in comparison to smelter or chemical processing. This method meets the parallel objectives of resource recovery and pollution mitigation; however, current practices of microbial leaching are inadequate to recover the critical metals belonging to precious groups [27,28].

As a matter of fact, in order to get an ideal separation result, different pretreatment techniques are included in these methods to obtain a mixture of metal and non-metal components [29]. Pretreatment methods include microwave irradiation, physicochemical method, pyrolysis, and so on. Pyrolysis is a pyrometallurgy technology based on incineration, is widely used to recover nonmetals from WPCBs, and has the advantages of simple equipment, high efficiency, and centralized treatment of toxic and harmful substances [30–32]. Typical pyrolysis techniques are summarized in Table 1.

Table 1. A tabular summarized data on pyrolysis.

Technique	Salient Features	Ref.
Rotary kiln	Large processing capacity	[33]
Fixed bed	Simple process equipment	[34,35]
Fluidized bed	Shorten pyrolysis time	[36]
Vacuum pyrolysis	Avoid secondary pyrolysis	[37]

However, applying pretreatment before mechanical crushing and physical/chemical separations will make the separation processes complicated and increase the cost. Furthermore, WPCBs are composed of 40% organics (epoxy resin) and 60% inorganics (glass fiber). The non-metallic components (NMC) of WPCBs are usually unable to be safely treated and effectively recovered due to low value and harmful substances [32,38]; therefore, new routes that are environmentally friendly, energy-saving, and involve resources that are renewable, should be subjected to the sustainable development WPCB recycling.

Thermal plasma technology is a process that demonstrates a high performance when processing different types of waste [39,40]. Thermal plasma can provide ultra-high temperature reaction conditions, and an adjustable oxidative, reductive, or inert gas atmosphere, which allows it, as a feasible technique, to implement the clean and highly efficient conversion of low-quality feedstock and hard-to-treat intermediates and wastes [41,42]. Most of the molecules have changed into atomic, ionic, or excited states under the plasma condition. These active groups provide high chemical activity, and create favorable conditions for the pyrolysis reaction. In a plasma-catalyst hybrid system, reactive plasma species can interact with the catalyst, which can influence the reaction pathways and the selectivity of the desired products [43–45]. Plasma catalytic conversion is usually used in non-thermal plasma technology.

In the present work, a three-phase AC arc plasma pyrolysis device was designed to treat WPCBs in order to solve the problem of the difficulties that occur when separating recyclable metal from the organic matrix directly and effectively by mechanical crushing. In addition, hydrogen will be introduced into the plasma system so that organics in WPCBs can be converted into combustible gas or other chemical raw materials for recycling upon plasma pyrolysis, with almost no pollution emission.

2. Plasma Device Design

The homemade plasma pyrolysis device consists of three electrodes with a three-phase AC power supplier connected to them. Figure 1 presents the illustration of the three-phase AC arc plasma device, including power supplier (4), gas supplier (2), cooling system (Not shown in the illustration), and sample collection system (3). Graphite rods (6) are used as electrodes, copper tubes (7) are used to connect the graphite electrodes with the power supplier, and polytetrafluoroethylene (Teflon, 8) is used for electrode jackets for insulation between the electrodes and reactor walls. The copper tubes (7) are processed into a hollow structure, into which circulating water can be introduced to cool the graphite electrode. Terminals (9) are used to ensure the stability of power supply.

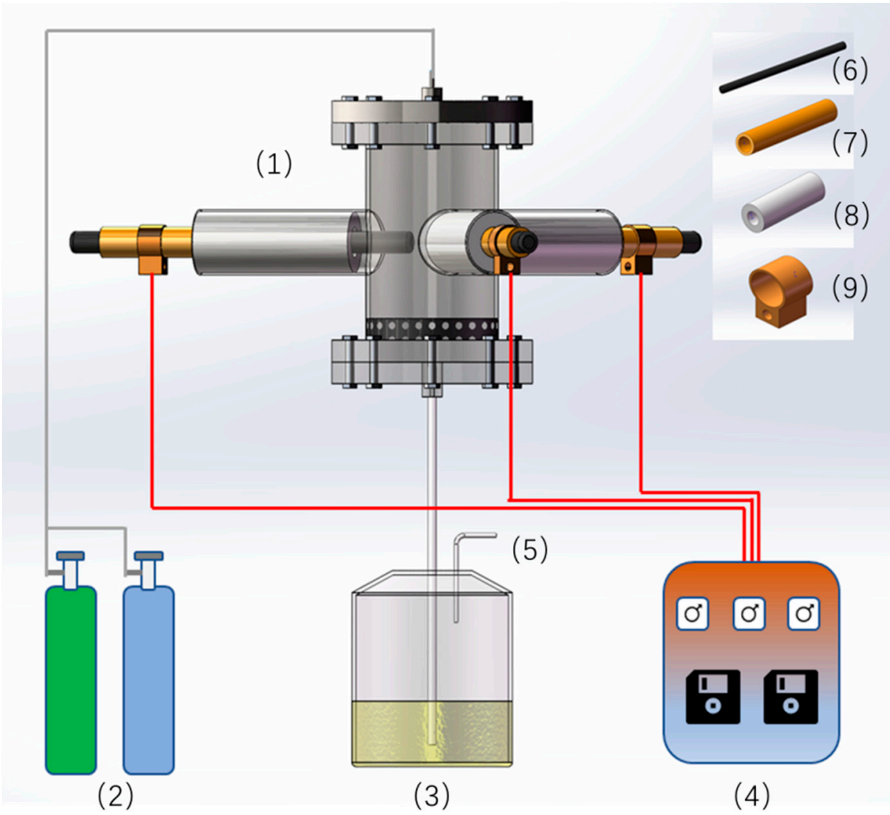
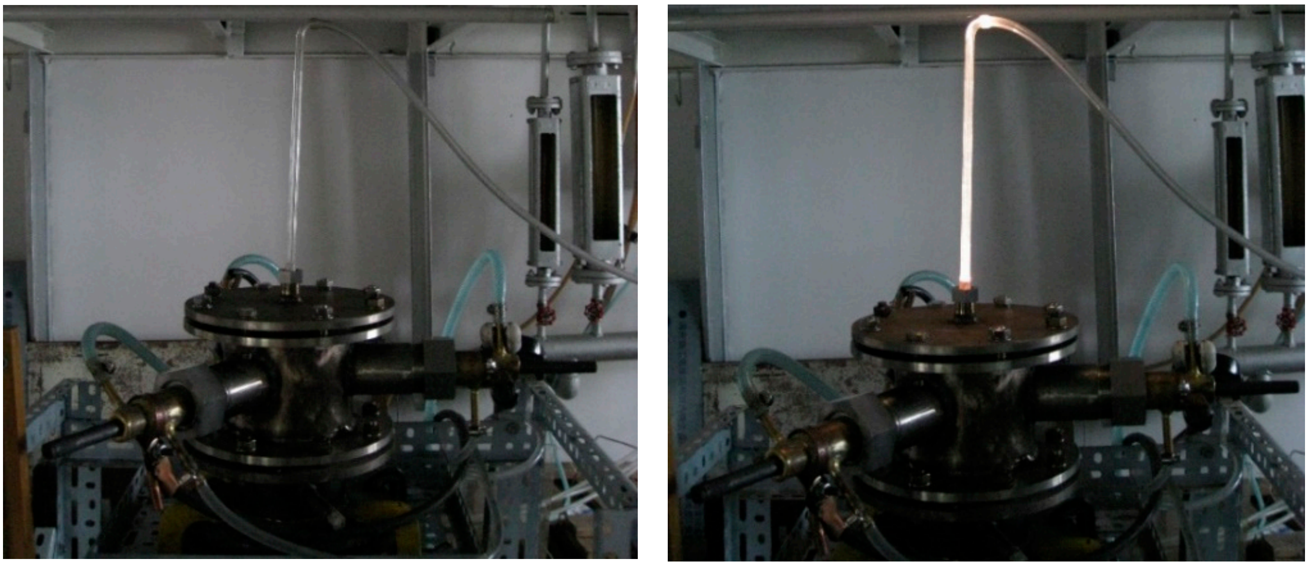


Figure 1. Illustration of the three-phase AC arc plasma device. (1) pyrolysis reactor; (2) gas supplier; (3) oil collector; (4) powder supplier; (5) gas collector; (6) graphite rod; (7) copper tube; (8) electrode jackets; (9) electrode connector.

The phase difference between the three electrodes is 120°. The temperature of the arc plasma can be adjusted by power input, and the atmosphere in the pyrolysis furnace can be controlled by introducing different gases, which can be oxidation atmosphere (O_2), inert atmosphere (Ar), or reduction atmosphere (H_2). The cooling system is used to cool the graphite electrodes, and their Teflon jackets prevents them from overheating. Both liquid and gaseous products are collected from the bottom of the reactor.

Plasma power supply equipment was provided by the Beijing Qinghe electric welding machine factory. Figure 2 presents the photos of the three-phase arc plasma setup before and after arc ignition. A steady three-phase AC arc plasma could be obtained in our laboratory, which overcame the problems of poor continuity and low energy of single-arc discharge. Compared with DC power supply, AC power supply avoided a rectification process and thus exhibited higher efficiency, otherwise, common electric welding machines were used as AC power supplier, which helps bring down the equipment purchase and maintenance costs. The discharge voltage between three-phase electrodes was higher than that reported in the literature, which could generate higher power and provide a higher pyrolysis temperature [46].



(a) (b)

Figure 2. Photos of the three-phase AC arc plasma setup before (**a**) and after (**b**) arc ignition.

The plasma could be directly ignited and maintained in air; however, it was discovered that N_2 and O_2 would react with each other to form NO_x, which produced an unpleasant smell. A flue gas analyzer (testo-335) was used to determine the concentration of NO_x in the exhaust gas, when an air compressor was used as the gas supplier. Collected data was summarized in Table 2.

Table 2. The concentration of NO_x in the exhaust gas.

Air Flow (L/min)	NO (ppm)	NO_2 (ppm)	NO_x (ppm)
1.0	1304	209	1513
1.5	1215	133	1348
2.0	1120	133	1253

It can be seen that NO_x exists in the exhaust gas, and the content of NO_x decreases with the increase of the air flow rate. The reason is that increasing the air flow rate may reduce the energy that can be obtained per unit gas, resulting in the reduction of NO_x content. NO_x is one of the main causes of environmental problems such as acid rain, ozone holes, and photochemical smog. Moreover, the organics in WPCBs would burn in the oxygen enriched plasma and lose their recycling value, and the metals would also be oxidized; therefore, Ar instead of air was used as plasma gas in the present work for protective recycling of different valuable metals from WPCBs.

The plasma gas flow has a great influence on the shape of three-phase AC arc plasma, which also provides a convenient condition for plasma pyrolysis. After being electrified, the electrodes discharge between each other and the arc of the plasma exhibits a triangular shape before the gas flow is introduced into the reactor. When Ar or N_2 continuously blows towards the center of the triangular, the plasma arc extends downwards. The transfer of the plasma arc will cause the high temperature center to leave the three-electrode region, which is conducive to the application of the plasma equipment in the pyrolysis of WPCBs.

A grid structure is designed to hold the WPCBs so that they can be pyrolyzed at the bottom of the reactor, which ensures the smooth passage and collection of gas and liquid products of plasma pyrolysis. The diameter of the reaction chamber is 300 mm, and the height is 400 mm, the outer layer of which is 1Cr18Ni9Ti alloy steel and the inner layer is graphite.

3. Experimental and Results

The process flow of three-phase AC arc plasma for recycling WPCBs is shown in Figure 3. WPCBs were added on the grid at the bottom of the reactor, in which organics in WPCBs would pyrolyzed into small molecule combustible gas and oil upon plasma heating. The adhesion between metal and glass in the solid residue would be destroyed completely, and effective separation by simple mechanical crushing can be carried out afterwards. Considering modern separation and purification methods, the recirculation of plasma gases, including H_2 and Ar, within the plasma reactor will make the present plasma recycling of WPCBs a more perfect technique.

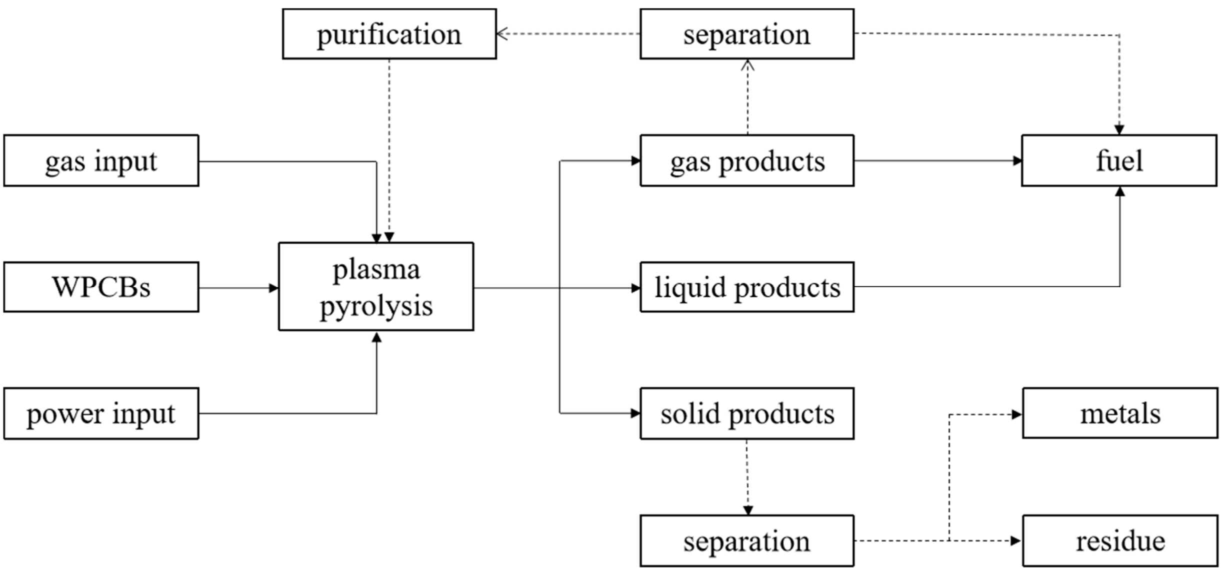


Figure 3. Scheme of the technological process in three-phase alternating arc plasma assisted utilization of WPCBs.

The specific experimental process is as follows: the WPCBs to be treated were placed in the plasma arc area, and plasma gas was supplied from the gas delivery system and sent through the gas inlet on the top of the reactor. After the atmosphere was balanced, the plasma arc was ignited by short-circuit discharge, and the pyrolysis of WPCBs began. The system pressure was shown using a U-shaped tube connected with the system and it was kept micro positive during the experiment. The pyrolysis products were led out along the corundum tube at the bottom of the reactor, the oil was collected in the wide mouth bottle after cooling, and the gas was collected by exhaust gas extraction.

Figure 4a shows the pictures of WPCBs. Before arc plasma pyrolysis, the components of waste printed circuit boards are so closely combined that it is difficult to effectively separate the available metal components from organic components and glass fibers. Figure 4d shows the solid residue after pyrolysis, which had a mass of 60% before pyrolysis. It can be seen that the combination between the printed metal wire and the substrate is very loose, because the organic adhesive was decomposed and the strong adhesion between the metals and fiberglass boards was destroyed. After simple coarse crushing, various separation methods can be used to recover the metal and non-metal components, and the separated metal can be further processed and purified. Figure 4c shows the picture of the liquid product collected from the bottom of the reactor. It is viscous and brown in color. As a matter of fact, it is so viscous that a lot of it attaches onto the inner wall of the pipe and collecting bottles. Figure 4b is the air bag containing the gas product, which is the most important concern of this study.

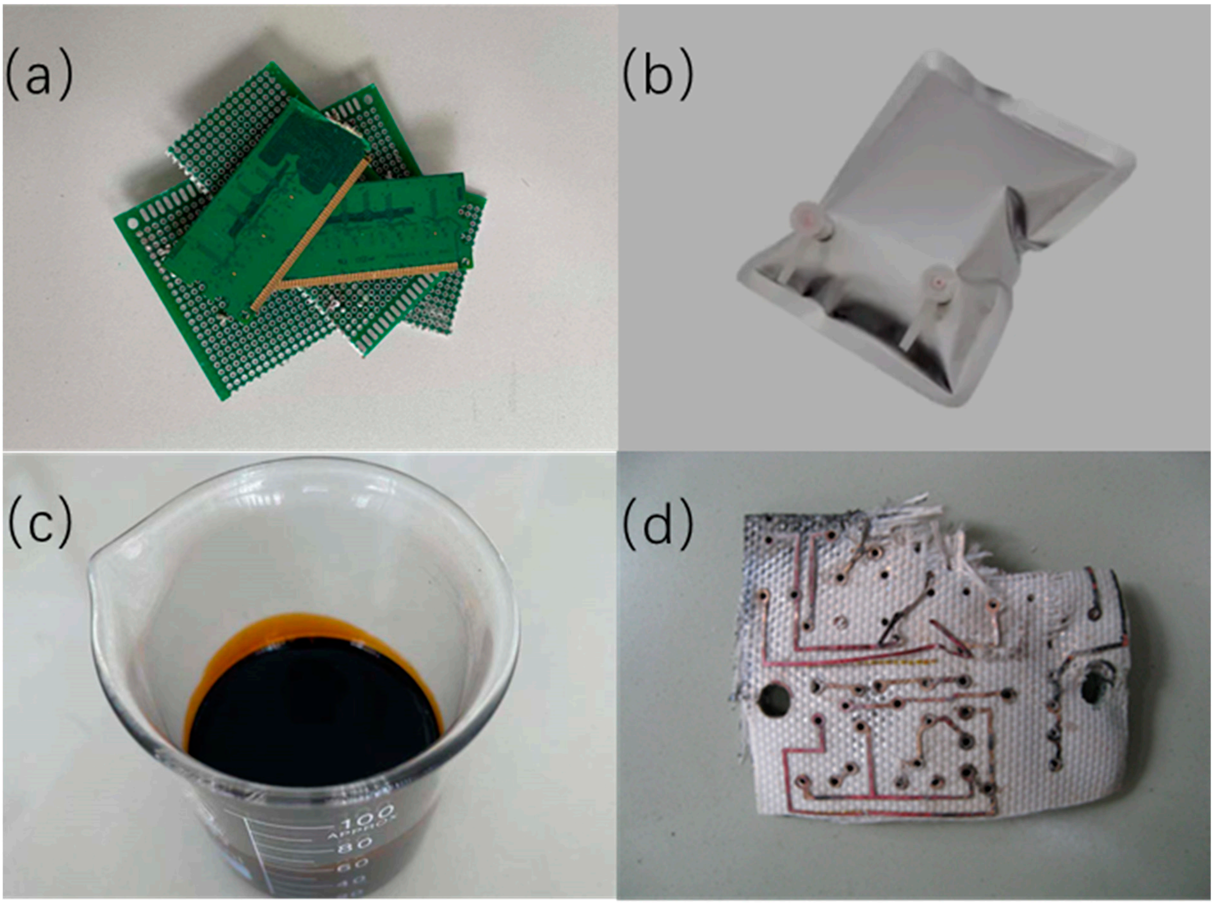


Figure 4. WPCBs (**a**) and the pyrolysis products in gas (**b**), liquid (**c**), and solid (**d**) phase.

Table 3 shows the composition and content of the gas product by Ar-plasma pyrolysis using Ar as a plasma working gas. All experiments were repeated three times, and the syngas sampling time lasted for 10 s, and from 30 s to 40 s during each experiment. Three samples as one group were collected in gas bags for each experiment. Every group of samples were characterized using a gas chromatograph-mass spectrometer (GCMS QP2010 Ulltra, Shimadzu, Kyoto, Japan). The mean value and standard deviation were calculated based on the test results of each group.

Table 3. Composition and content of gas product by Ar-plasma pyrolysis.

No.	1	2	3	4	5
Composition	H_2	CO_2	CO	CH_4	C_2H_4
Content	36.18%	35.55%	21.84%	3.56%	2.87%
Standard deviation	0.17%	0.14%	0.23%	0.12%	0.11%

It can be seen that the main species of pyrolysis gas product by Ar-plasma are H_2, CO_2 and CO. This result is inconsistent with that reported in the literature [47]. H_2, CO_2, and CO account for 36.18%, 35.55%, and 21.84%, respectively. In addition, CH_4 and C_2H_4 account for 3.56% and 2.87%, and no other small molecules of combustible hydrocarbons are detected.

Thermal plasma exhibits novel characteristics such as high temperature and high chemical activity, which can promote different decomposition reactions or even initiate reactions that cannot occur under normal pyrolysis conditions. That is the reason why Ar-plasma can destroy the strong adhesion between the metals and fiberglass boards and decompose the macromolecule solids into liquids and gases with small molecules.

In order to improve the gas quality and make more use of the organics in the WPCBs, H_2 is mixed to the plasma gas, to form an Ar/H_2-plasma. It is supposed that active H species have more obvious effects on the cleavage of organic macromolecules. Accurately calculating the oil productivity based on the collection of all the liquid products is complicated, as they were viscous, and a lot of them attached onto the inner wall of the pipe and collecting bottles; therefore, we focus on the examination of the gas products. Table 4 shows the composition and content of gas product by Ar/H_2-plasma pyrolysis. The mean value and standard deviation were calculated based on the test results of each group.

Table 4. Composition and content of gas product by Ar/H_2-plasma pyrolysis.

No.	1	2	3	4	5	6
Composition	H_2	CO_2	CO	CH_4	C_2H_4	C_3H_6
Content	93.53%	2.26%	1.87%	0.72%	1.57%	0.04%
Standard deviation	0.09%	0.11%	0.07%	0.05%	0.04%	0.005%

When hydrogen was used as one of the plasma gases, C_3H_6 appeared in the products of Ar/H_2-plasma pyrolysis in addition to H_2, CO_2, CO, CH_4, and C_2H_4. This is the most obvious difference between the Ar/H_2-plasma and Ar-plasma pyrolysis process. Furthermore, the content of H_2 accounts for 93.53%, which is much higher than other species. The reason for the abnormal growth was that hydrogen, as a plasma gas, was trapped in the product.

In order to study the influence of plasma gas change on the formation of other combustible small molecule gases, we ignore the hydrogen content in the product and investigate the percentage changes of other components only. Figure 5 shows a columnar comparison of the pyrolysis product content of Ar-plasma and Ar/H_2-plasma. It can be seen clearly that the content of carbon dioxide in the product decreases significantly from 55.7% to 35.0%, the content of carbon monoxide in the product decreases slightly from 34.2% to 29.0%, whereas other species and the content of combustible small molecular hydrocarbons increase. CH_4 increases from 5.6% to 11.2%, C_2H_4 increases from 4.5% to 24.3%, and C_3H_6 increases from 0% to 0.5%.

As mentioned above, thermal plasma has a high thermal performance, and its temperature can be as high as 10^4 K, which is much higher than the heating process or metallurgical and chemical reaction process of ordinary pyrolysis. At the same time, it also has a high thermal conductivity and temperature gradient. Under the conditions of thermal plasma, most of the molecules have changed into atomic, ionic, or excited states. These active groups cause the plasma to have a high chemical activity, and they create favorable conditions for the pyrolysis reaction. In our previous work, in situ optical emission spectroscopy (OES) was carried out to diagnose and determine the active particles in Ar/H_2-plasma, and H_α and H_β were detected in the emission spectrum, indicating that there were highly active H radicals in Ar/H_2-plasma which can be used as a highly active reducing medium in chemical reactions [47,48]; therefore, Ar/H_2-plasma can provide highly chemically active hydrogen and break chemical bonds in organic macromolecules to produce small molecules of combustible gases.

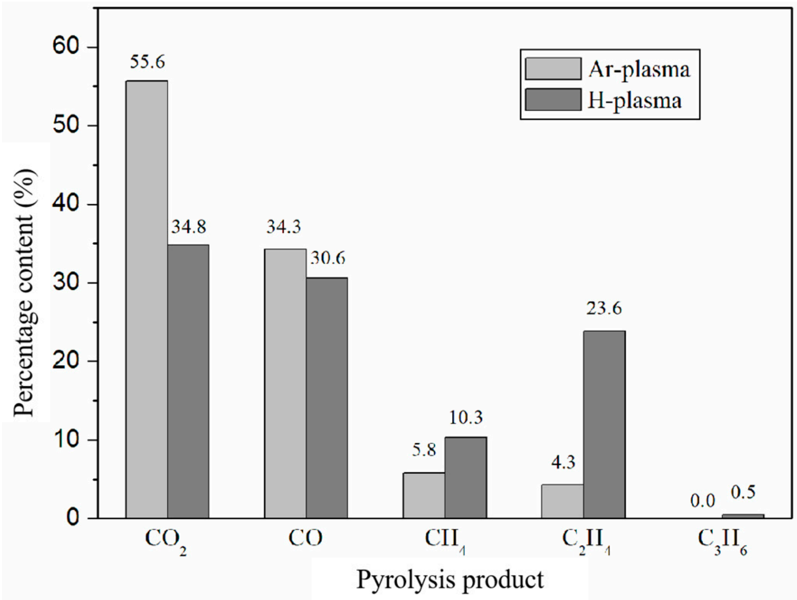


Figure 5. Columnar comparison of the pyrolysis product content of Ar-plasma and Ar/H_2-plasma.

In order to further study the important role of Ar/H_2-plasma in pyrolysis, different hydrogen flow rates were investigated when the pyrolysis if WPCBs was conducted using the three-phase AC arc plasma. Figure 6 shows the effect of the hydrogen flow rate on the percentage of the gas product. With the increase of the hydrogen flow rate in the arc plasma, the composition of gas products obviously changes. The production of C_2H_4 increases significantly, whereas the volume fraction of the non-combustible component CO_2 gradually decreases and disappears.

As reported in the literature, the active species in the Ar/H_2-plasma participates in the pyrolysis process of circuit boards, which promotes the pyrolysis process of long-chain hydrocarbons [49]. The increase in the hydrogen flow rate provided a higher concentration of active species in the plasma arc, which could contribute to a change in the volume fraction of CO_2 and hydrocarbons.

Electric energy was supplied by three electric welding machines. Table 5 shows the typical equipment parameters.

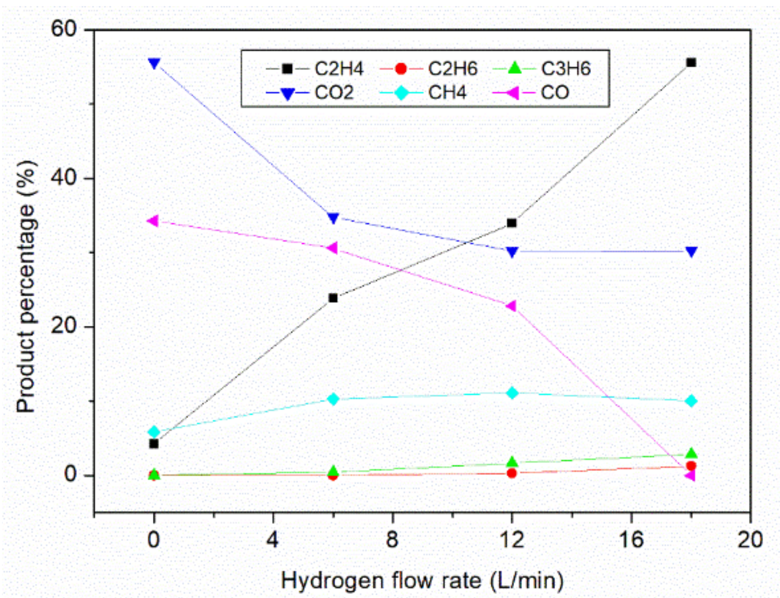


Figure 6. Effect of hydrogen flow rate on the percentage of the gas product.

Table 5. Typical equipment parameters.

No.	Parameters	Value		
1	Welding current range		50 A/22 V~250 A/30 V	
2	Rated welding current	250 A	194 A	148 A
3	C onventional load voltage	30 V	27.8 V	25.9 V
4	Rated maximum input current		41 A	
5	Rated input voltage		380 V	

The plasma power can be adjusted by setting welding current. Figure 7 shows the effect of current on the percentage of the gas product with the hydrogen flow rate at 6 L/min. The change trend caused by the current is similar to that caused by the hydrogen flow. The current increase leads to the powder increase, and further improves the plasma enthalpy and the ionization degree of hydrogen. The designed laboratory plasma pyrolysis device is a discontinuous fixed bed reactor, in which the heat will accumulate and cause the temperature to rise; therefore, compared with the influence of temperature change, the ionization degree of hydrogen might have a greater effect on the pyrolysis results. That is the reason why the change trend caused by current and hydrogen flow alteration is similar.

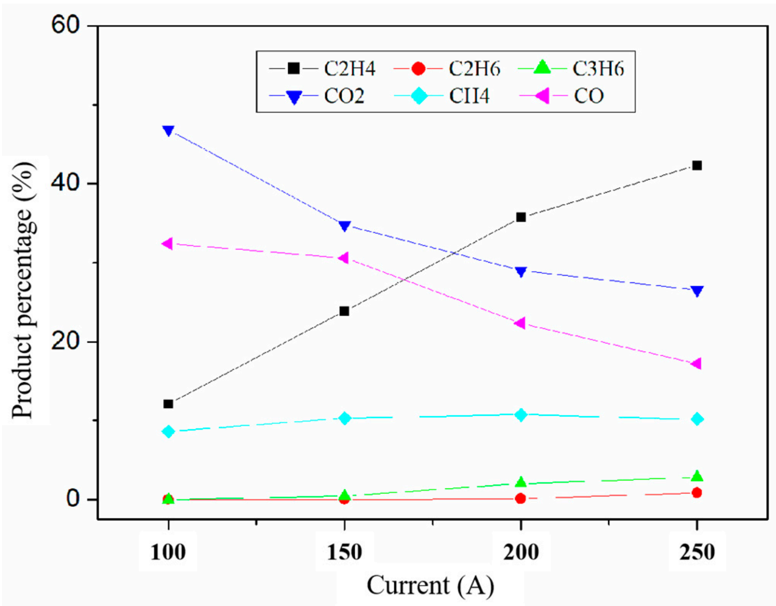


Figure 7. Effect of current on the percentage of the gas product.

4. Conclusions

A three-phase AC arc plasma pyrolysis device was designed for the resourcing treatment of WPCBs. Several conclusions can be drawn from the results and discussion:

(1) The three-phase AC arc plasma can operate stably, which overcomes the problems of poor continuity and low energy of single-arc discharge.
(2) Air-plasma would generate NO_x contaminants, burn the organics, and oxidize the metals, and thus, one would have had to resort to other gases.
(3) Ar-plasma can break the long chains of organic macromolecules to make combustible gas.
(4) Ar/H_2-plasma promoted the decrease of carbon dioxide and increase of combustible small molecular hydrocarbons in the pyrolysis product.
(5) The strong adhesion between the metals and fiberglass boards would be destroyed by plasma pyrolysis and facilitate subsequent separation.
(6) This laboratory work provides a novel three-phase AC arc plasma device and a new way of recycling WPCBs with high value.

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