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Remelting of Copper and Its Waste in a Controlled Atmosphere

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Dedication

I dedicate this work

To my wonderful parents Djamel and Fatna for their love and support

To all the members of my family

To all friends and classmates for their constant support

To all the teachers who have passed me by in my academic journey

Acknowledgement

This work could not have been accomplished without the help of God and His will. Praise to God always and for everything

My sincere and respectful thanks to my supervisor Dr. MESSAOUDI Salim for his advice and for his help in completing this work.

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All the person who helped me, near or far, in the accomplishment of this modest work will find here a sign of gratitude and thanks.

Summary

General Introduction

With the decrease in world reserves of minerals and more specifically metals. The metallurgical industries, which constitute the main and efficient axis for the extraction and processing of metals, are directly linked to this global situation. In this case, many countries and companies find themselves forced to review their policy of self-sufficiency for many types of minerals and especially the most essential such as aluminum and copper. Copper is one of the most consumed metals in the world; and mainly it is the electrical energy transport sector which is the main user of this metal. But due to strong global demand and the cost generated by the extractive metallurgy of copper, its courses have soared during the last five years. In addition to the price, copper mining leaves many side effects that directly affect the environment and destroy nature. In an effort to overcome these problems, the world has turned to the strategy of recycling scrap copper.

Biskra Cable Industries Company (ENICAB-BISKRA) is a company specializing in the manufacture of various types of electric wires and cables, which is located in Biskra. As it imports its raw materials (copper rod and others), it has been directly affected by this global copper crisis. It therefore began to work on ways of recycling copper in order to maintain its economic objectives. It is in this context that this work is born and is proposed to us by the company. The company wishes to exploit three types of copper sources; the cathode (ETP) and two scrap copper. Our job is to find the effective mixture between the three types and study their chemical and structural properties.

So, this study is therefore a preliminary contribution made at the request of the company to help it in its orientation towards the good choice of copper waste which will constitute its raw material.

The characterization techniques used are as follows:

- chemical analysis
- Optical microscopy.

The dissertation is made up of three chapters:

- Chapter I: Recycling and Metals Recycling
- Chapter II: Copper and Copper Alloys Recycling
- Chapter II: Experimental Procedure and Results

RECYCLING AND METALS RECYCLING

Introduction

Since some decades, waste recycling has become a main axis of sustainable development. In this chapter, we will review the notion of recycling by shedding the light on a group of elements related to it, such as the history and the origin, its principles, and the materials that can be used in recycling. We will focus on non-ferrous metals (aluminum, copper, zinc ...) and their recycling technologies and their applications in various industries

1. History

Although the recycling process as we know it only began about 40 years ago, the concept of reusing materials has been around for thousands of years.

The idea of waste before the industrial revolution was very different than our current idea of waste. Waste only included organics; wood, ash, textiles, and food waste. Fabrics, broken tools, pottery, and furniture was repaired and repurposed as many times as possible. The idea that something was broken and therefore garbage, is a new school idea as a result of our consumer society.

Until the end of the middle ages, the non-compostable waste was thrown into the street where it piled up or brought to a dump site where it was thrown in a pit and covered in soil once full. Waste was commonly burned as well.

Throwing trash into the streets worked for a few centuries until it proved to be a hazard in the middle ages. The decomposing trash lining the streets attracted rats, which aided in spreading the bubonic plague which killed an estimated 25 million people. In 1354, King Edward III implemented ‘rakers’; people who were hired to remove trash from the streets on a weekly basis. These rakers brought the waste to the Thames River to dump it.

In 1388, Britain outlawed the disposal of waste in public waterways and ditches.

Fast forward to 1864, health officials in Tennessee were aware of the correlation between the Yellow Fever epidemic and the garbage rotting around the public streets. The public was then urged to take their garbage to designated spots on the outskirts of town – similar to current day landfills. At this point, waste disposal was less about being sustainable and more about lowering the health risks to the public. The majority of the waste in these days was still organic, as plastic and excessive packaging had yet to be created and mass produced.

In 1897, New York City opened one of the first Material Recovery Facilities. Valuable materials from the trash were brought there, sorted, and then reused by the public. In-demand materials of the day included rubber, burlap, and even horse hair. In the early 20th century, pop manufacturers began providing deposit returns for those who brought back their empty bottles. Some bottles were making up to twenty trips back and forth because people wanted their five cent deposit back.

This reinforced the idea that products – namely containers – can be reused a number of times. In addition, the material had value. This lessened the cost to the manufacturers and incentivized the recycling process to the general public. The idea of reusing old material instead of using raw materials to create products was a revolutionary way to save time, money, and energy.

During the Second World War, tin cans, vinyl records, scrap metal, and rubber were some of the most popular items requested by the government. These items were bought up and used to create ammunition and other materials to assist the government with the war.

In the mid-20th century, the focus of recycling changed from a way to save money, to a way to reduce waste and maximize the lifetime of a material **[7].**

2. Definition and Principe

By definition, recycling is the process of collecting and processing materials, that would otherwise be thrown away as trash, and turning them into new products **[10].**

Thus the recycling notion consists of the recovery and reprocessing of waste materials for use in new products. So, the basic phases in recycling are the collection of waste materials, their processing or manufacturing into new products, and the purchase of those products, which may then themselves be recycled.

Typical materials that are recycled include iron and steel scrap, aluminum cans, glass bottles, paper, wood, and plastics.

The materials reused in recycling serve as substitutes for raw materials obtained from such increasingly scarce natural resources as petroleum, natural gas, coal, mineral ores, and trees. Recycling can help reduce the quantities of solid waste deposited in landfills, which have become increasingly expensive. Recycling also reduces the pollution of air, water, and land resulting from waste disposal **[11].**

Figure I.1: Plastic, glass, and metal containers in a recycling bin **[11].**

3. Types of recycling operations

There are two broad types of recycling operations: internal and external.

– Internal recycling is the reuse in a manufacturing process of materials that are a waste product of that process. Internal recycling in the other hand is common in the metals industry, for example the manufacture of copper tubing results in a certain amount of waste in the form of tube ends and trimmings; this material is remelted and recast. Another form of internal recycling is seen in the distilling industry, in which, after the distillation, spent grain mash is dried and processed into an edible foodstuff for cattle.

– External recycling is the reclaiming of materials from a product that has been worn out or rendered obsolete. An example of external recycling is the collection of old newspapers and magazines for repulping and their manufacture into new paper products. Aluminum cans and glass bottles are other examples of everyday objects that are externally recycled on a wide scale.

Society’s choice of whether and how much to recycle depends basically on economic factors. Conditions of affluence and the presence of cheap raw materials encourage human beings’ tendency to simply discard used materials. Recycling becomes economically attractive when the cost of reprocessing waste or recycled material is less than the cost of treating and disposing of the materials or of processing new raw materials **[11].**

4. Some Materials That Can Be Recycled

a. Rubber:

Though much used rubber was formerly burned, this practice has been greatly curtailed in most countries in order to prevent air pollution. Internal recycling is common in most rubber plants; the reprocessed product can be used wherever premium-grade rubber is not needed. External recycling has proved a problem over the years, as the cost of recycling old or worn-out tires has far exceeded the value of the reclaimed material. Shredded rubber can be used as an additive in asphalt pavements, and discarded tires may be used as components of swings and other assorted recreational climbing equipment in “tire playgrounds” for children**[17].**

b. Paper and Other Cellulose Products:

Paper is well known for its recycling potential, and is often seen as the recycling industry’s biggest success story. Paper lends itself to be broken down back to cellulose fibers, which can be used as building blocks for the production of recycled paper. These cellulose fibers can however not be recycled endlessly. During the recycling process, the fibers are shortened, until ultimately they become too short to apply in new paper products. Theoretically, the same fibers can be recycled up to 6 or 7 times before fiber properties have diminished to such extend that they cannot be used for paper anymore. These short cellulose fibers end up in what is called paper sludge **[16].**

c. Glass:

ing glass is one of the many ways we can help reduce pollution and waste. Every day we throw away tones of rubbish and glass is a significant part of it. Instead of letting landfills pile up with glass objects that are a threat to safety and the environment, we can use it again.

Glass can be melted down and made into many different forms from drinking glasses to glass fiber. When the glass is taken to a manufacturing or recycling plant, it is broken up into smaller pieces called cullet.

The broken pieces are crushed, sorted, cleaned, and prepared to be mixed with other raw materials like soda ash and sand. The raw materials and glass pieces are melted in furnace and then shaped into mould to make new bottles of different colors and sizes. New recycled bottles and jars are made in this way **[18].**

d. Plastics:

Plastics account for almost 10 percent by weight of the content of municipal garbage. Plastic containers and other household products are increasingly recycled, and, like paper, these must be sorted at the source before processing. Various thermoplastics may be remelted and reformed into new products.

Thermoplastics must be sorted by type before they can be remelted. Thermosetting plastics such as polyurethane and epoxy resins, by contrast, cannot be remelted; these are usually ground or shredded for use as fillers or insulating materials. So-called biodegradable plastics include starches that degrade upon exposure to sunlight (photo degradation), but a fine plastic residue remains, and the degradable additives preclude recycling of these products.

e. Construction and Demolition Waste:

Construction and demolition (C&D) debris (e.g., wood, brick, portland cement concrete, asphalt concrete and metals) can be reclaimed and reused to help reduce the volume taken up by such materials in landfills. Concrete debris consists mostly of sand and gravel that can be crushed and reused for road subcase gravel. Clean wood from C&D debris can be chipped and used as mulch, animal bedding, and fuel. Asphalt can be reused in cold-mix paving products and roofing shingles. Recovered wallboard can be used as cat litter. As landfill space becomes more expensive, more of these materials are being recycled.

f. Domestic Refuse:

Domestic refuse (municipal solid waste) includes garbage and rubbish. Garbage contains highly decomposable food waste (e.g., kitchen scraps), while rubbish is the dry, non-putrescible component of refuse. Once glass, plastics, paper products, and metals have been removed from domestic refuse, what remains is essentially organic waste. This waste can be biologically decomposed and turned into humus, which is a useful soil conditioner, and kitchen scraps, when decomposed with leaves and grass in a compost mound, make an especially useful soil amendment. These practices help reduce the amount of material contributed by households to landfills.

g. Wastewater:

Treated wastewater (domestic sewage) can be reclaimed and reused for a variety of purposes, including golf course and landscape irrigation. With achievement of appropriate (secondary) treatment levels, it may be reused for the irrigation of certain agricultural crops. After very high levels of advanced (or tertiary) treatment and purification, it may even be used to supplement drinking water supplies.

However, because of public resistance to the direct reuse of treated sewage for domestic purposes, recovered water must be recycled indirectly. This is done by injecting it into the ground or storing it in ponds and allowing it to seep into naturally occurring aquifers so that it is further purified as it slowly moves through the geologic strata. In some regions of the world where water supplies are inadequate because of recurring drought and rapidly expanding populations, the recycling and reuse of treated wastewater is a virtual necessity. See wastewater treatment **[11].**

5. Metal Recycling

Scrap metal recycling is a process as well as being the basis for a powerful industry. Scrap metal recycling involves the recovery and processing of scrap metal from end-of-life products or structures, as well as from manufacturing scrap, so that it can be introduced as a raw material in the production of new goods. It can be recycled repeatedly with no degradation of its properties. It provides the raw material for new products, while offering a much lower carbon footprint and more efficient utilization of resources than new material.

Aside from environmental benefits, metal recycling is an extremely powerful economic activity. In 2015, the U.S. ferrous scrap industry was worth $18.3 billion. In 2014, U.S. nonferrous scrap had a value approaching $32 billion.

When talking about scrap metal recycling, it is important to differentiate between the two main categories of scrap metal: ferrous metal, and nonferrous metal. While ferrous metal contains some degree of iron (and in fact, its name is derived from the Latin term meaning iron), non-ferrous metal does not contain iron as a component. Nonferrous scrap includes aluminum, copper, lead, nickel, tin, zinc and others **[1].**

Figure I.2: A heap of scrap metal **[11].**

5.1.Ferrous Metals

Ferrous products (i.e., iron and steel) can be recycled by both internal and external methods. Some internal recycling methods are obvious. Metal cuttings or imperfect products are recycled by remelting, recasting, and redrawing entirely within the steel mill. The process is much cheaper than producing new metal from the basic ore. Most iron and steel manufacturers produce their own coke. By-products from the coke oven include many organic compounds, hydrogen sulfide, and ammonia. The organic compounds are purified and sold. The ammonia is sold as an aqueous solution or combined with sulfuric acid to form ammonium sulfate, which is subsequently dried and sold as fertilizer.

In the ferrous metals industry there are also many applications of external recycling. Scrap steel makes up a significant percentage of the feed to electric arc and basic oxygen furnaces. The scrap comes from a variety of manufacturing operations that use steel as a basic material and from discarded or obsolete goods made from iron and steel. One of the largest sources of scrap steel is the reprocessing of old automobile bodies.

Salvage operations on automobiles actually begin before they reach the reprocessor. Parts such as transmissions and electrical components can be rebuilt and resold, and the engine block is removed and melted down for recasting. After being crushed and flattened, the automobile body is shredded into small pieces by hammer mills. Ferrous metals are separated from the shredder residue by powerful magnets, while other materials are sorted out by hand or by jets of air. Only the plastics, textiles, and rubber from the residue are not reused. The same basic recovery procedures apply to washing machines, refrigerators, and other large, bulky steel or iron items. Lighter items such as steel cans are also recycled in large numbers **[11].**

5.2.Non-Ferrous Metals

The most commonly used non-ferrous metals are aluminums, copper, lead, zinc, nickel, titanium, cobalt, chromium and precious metals. Millions of tons of nonferrous scrap are recovered annually and used by smelters, refiners, ingot makers, foundries, and other manufacturers. Secondary materials are essential to the industry's survival because even new metals often require the combined use of recycled materials **[2].**

New metals made using recycled material

Copper > 40%, Lead > 35%, Aluminum> 33%, Zinc > 30%.

5.2.1. Recycling Processes

The metal recycling industry has an efficient structure with numerous small companies purchasing scrap material and feeding this to highly effective larger international businesses.

Non-ferrous metal recycling involves some, or all of the following steps:

a) **Sorting:** In order to be recycled appropriately, different types of non-ferrous metals need to be separated from each other, as well as from other recyclables such as paper and plastic.
b) **Baling:** Non-ferrous materials are compacted into large blocks to facilitate handling and transportation.
c) **Shearing:** Hydraulic machinery capable of exerting enormous pressure is used to cut metals into manageable sizes.
d) **Media separation:** Shredders incorporate rotating magnetic drums to separate non-ferrous from ferrous metals. Further separation is achieved using electrical currents, high-pressure air flow and liquid floating systems. Further processing may be needed.
e) **Melting:** The recovered materials are melted down in a furnace, poured into casters and shaped into ingots. These ingots are either used in the foundry industry or they can be transformed into flat sheets and other wrought products such as tubing, which are then used to manufacture new products **[2].**

Figure I.3: Feeding recovered copper wire into a conveyor belt for recycling **[2]**

Figure I.4: Baled zinc prepared for transport to a refinery **[2]**

5.2.2. Applications

All metals can be recycled with minimal or no loss of their original physical properties. They are such versatile materials that the possible applications for each metal and their combinations are endless **[2].**

a) Aluminum

The recyclability of aluminum is unparalleled. When recycled there is no degradation in properties when recycled aluminum is compared to virgin aluminum. Furthermore, recycling of aluminum only requires around 5 percent of the input energy required to produce virgin aluminum metal.

The properties of the various aluminum alloys have resulted in aluminum being used in industries as diverse as transport, food preparation, energy generation, packaging, architecture, and electrical transmission applications.

Depending upon the application, aluminum can be used to replace other materials like cooper, steel, zinc, tin plate, stainless steel, titanium, wood, paper, concrete and composites **[19].**

b) Zinc

Most zinc is used to galvanize other metals, such as iron, to prevent rusting. Galvanized steel is used for car bodies, street lamp posts, safety barriers and suspension bridges.

Large quantities of zinc are used to produce die-castings, which are important in the automobile, electrical and hardware industries. Zinc is also used in alloys such as brass, nickel silver and aluminum solder.

Zinc oxide is widely used in the manufacture of very many products such as paints, rubber, cosmetics, pharmaceuticals, plastics, inks, soaps, batteries, textiles and electrical equipment. Zinc sulfide is used in making luminous paints, fluorescent lights and x-ray screens **[20].**

c) Lead

The most important commercial use of lead is in the manufacture of lead acid storage batteries. It is also used in alloys such as fusible metals, anti-friction metals, welding and type metal. Lead is used to cover cables and is widely used in plumbing. Due to its excellent vibration dampening properties, and is used to support heavy machinery **[21].**

d) Tin

Tin's more modern application is as a solder for the electronics industry. Used in various purities and alloys (often with lead or indium), tin solders have a low melting point, which makes them suitable for bonding materials.

Tin alloys can also be found in a wide variety of other applications, including Babbitt bearings (often alloyed with copper, lead, or antimony), automobile parts (alloyed with iron), dental amalgams (alloyed with silver), and aerospace metals (alloyed with aluminum and titanium). Alloys of zirconium (often referred to as Zircaloys), used in nuclear reactors, also often contain a small amount of tin **[22].**

e) Copper

After silver, copper has the best electrical conductivity of all the elements. It is also very worthy thermal conductor and is readily alloyed with other metals such as lead, tin and zinc for foundry applications to produce, among other goods, products for the transmission of water such as valves. Other common applications for recovered copper include:

Electrical applications: Wires, circuits, switches and electromagnets.

Piping: Plumbing fittings, refrigeration, air conditioning and water supply systems.

Roofing and insulation.

Household items: cookware, doorknobs, and cutlery **[2].**

5.2.3. Recycling Facts

- Almost 40% of the world's demand for copper is met using recycled material.
- At present, approximately 30% of global zinc production comes from secondary zinc.
- Over 80% of the zinc available for recycling is eventually recycled **[2].**

a) Aluminum

- 75% of the aluminum ever produced is still being used in some form today. Most of this aluminum is used in building applications.
- The recycling process uses only 5 per cent of the energy used in the primary production of aluminum. This means recycling aluminum saves 95% of the energy used to produce virgin aluminum.
- Recycling a single aluminum can save enough energy to run a television for up to 3 hours. It also avoids CO2 emissions equivalent to a 1-mile car journey, and could be back on the supermarket shelf as another can within 60 days.
- Aluminum can be recycled indefinitely without loss of quality, unlike materials such as plastic, paper and timber that are usually down-cycled, reducing quality and limiting their use every time.
- Two of the world's largest aluminum producers, Canada and Norway, use 100% hydropower as an energy source. Aluminum refineries in Launceston and Invercargill, NZ are also powered by hydropower.
- Aluminum recycling over a year saves enough electrical energy to power The Netherlands for a year. It also saves more than 90 million tons of CO2 each year.
- Emission savings from aluminum recycling are increasing all the time, having doubled since 1990 and are expected to increase by a further 50% by 2020 **[23].**

b) Zinc

- Zinc is 100% recyclable. Over 80% of the zinc available for recycling is currently recycled.
- More than one-third of the zinc consumed in North America is produced from recycled materials **[25].**

c) Lead

- In 2020, about 12.4 million tons of refined lead was produced, including from primary or mined sources and secondary sources, according to Wood Mackenzie data.
- About two-thirds of refined lead is produced through recycling.
- About 86% of refined lead is used in lead acid battery production in the world, according to estimates by ILZSG **[24].**

d) Tin

- Globate tin production amounts to 350,000 tons of which 50,000 tones is produced from scrap and other secondary sources.
- Primary production of tin requires 99% more energy than secondary production **[2].**

e) Copper

- Copper's recycling value is so high that premium-grade scrap holds at least 95% of the value of the primary metal from newly mined ore.
- Recycling copper saves up to 85% of the energy used in primary production.
- In order to extract copper from copper ore, the energy required is approximately 95 million Btu/ton. Recycling copper uses much less energy, about 10 million Btu/ton.
- By using copper scrap, we reduce CO2 emissions by 65% **[2].**

Conclusion

As a conclusion to this chapter, we can deduce that recycling of metals helps us to make sustained use of them. It conserves natural resources, energy, therefore reduces pollution and protects nature. Due to the unique and valuable properties metals, their recycling will remain an integral part of future industrial society**.**

COPPER AND COPPER ALLOYS RECYCLING

Introduction

Copper conducts electricity and heat very well which makes the metal an essential raw material in building communities and modern economies; 65 per cent of all copper produced around the world today is used to generate and conduct electricity. It is an incredibly important metal in the transition to alternative energy sources such as solar, wind and hydroelectric that needs large amounts of copper both to generate electricity and transfer it over long distances with as little energy loss as possible. The supply of copper is thus one of the most important factors for effective energy transition **[4].**

In this chapter, we will shed light on the history of copper and its properties that made it one of the most important metals for consumption and use in various industries and methods of its production. We will focus on its recycling, its economic goals and the technologies needed to carry it out.

1. History

Copper has been in use by civilization for over 10,000 years and has been recycled since early times. Due to the fact that the copper does not degrade during recycling, its use today could have been first fabricated into objects thousands of years ago. Copper is highly prized by scrap metal collectors and scrap metal recycling businesses. The nonferrous metal is the best conductor of electricity except for silver. That electrical and thermal conductivity; along with properties of high ductility and malleability, make it one of the most demanded metals by industry, eclipsed only by iron and aluminum.

Copper has been used and recycled by people for over10,000 years, with a pendant dated to approximately 87,000BC have been found in what is now northern Iraq. Around 8000 B.C., copper emerged as a substitute for stone, and by 4000 B.C., Egyptians were heating and casting copper into shapes. Around 3500 B.C., the technology of copper processing continued to grow as the process of smelting ores was discovered, harkening the introduction of the Bronze Age.

The Mediterranean island of Cypress was the source of copper used by the ancient Romans. They called the highly coveted ore “aes Cyprium,” which translates into English as "metal of Cyprus." This name was shortened to cyprium, and later, cyprium was changed to cyprium, this latter term was the genesis of the English word, "copper" **[5].**

2. Background Information On Copper and Copper Alloy Scrap

2.1.Copper: definition and scope

Copper is a malleable and ductile metallic element that is an excellent conductor of heat and electricity as well as being corrosion resistant and antimicrobial. It occurs naturally in the earth's crust in a variety of forms. It can be found sulphide deposits (as chalcopyrite, bornite, chalcocite, covellite), in carbonate deposits (as azurite and malachite), in silicate deposits (as chrysocolla and dioptase) and as pure 'native' copper. As trace element, copper also occurs naturally in humans, animals and plants.

Copper can be alloyed with other metals, such as zinc (to form brass), aluminum or tin (to form bronzes), for use in specialized applications. Bronze is a mix of copper that contains as much as 25% tin. Brass is a mix of copper that contains between 5% and45% zinc. Small amounts of manganese, aluminum, and other elements may be added to bronzes and brasses to improve machinability, corrosion resistance, or other properties **[8].**

- Copper
- Miscellaneous Copper alloys (max. 5% alloy elements)
- Miscellaneous Copper alloys (over 5% alloy elements)
- Copper-aluminium alloys
- Copper-nickel alloys
- Copper-nickel-zinc alloys
- Copper-tin alloys, binary
- Copper-zinc-lead alloys
- Copper-zinc alloys, complex

2.1.1 Copper production

Copper results from two sources:

1. Primary production: extraction and processing (refining) of the raw mined material
2. Secondary production, originating from:

- Direct melt of 'new scrap' (waste resulting from the manufacturing process)
- Recycling of end-of-life products, using 'old scrap'.

Primary copper production can rely on two processes.

The first process is based on the extraction of copper-bearing ores. After the ore has been mined, it is crushed and ground followed by a concentration by flotation. The obtained copper concentrates typically contain around 30% copper. In the subsequent smelting process, copper is transformed into a 'matte' containing 50-70% copper. The molten matte is processed in a converter resulting in a blister copper of 98.5-99.5% copper content. In the next step, the blister copper is fire refined in the traditional process route, or, increasingly, re-melted and cast into anodes for electro-refining. The output of electro-refining is refined copper cathodes, assaying over 99.99% of copper.

Another important source of raw material is copper scrap. This scrap from either metals discarded in semis fabrication, finished product manufacturing processes ('new scrap') or obsolete end-of-life products ('old scrap'). Upon utilization of scrap, the refining process is referred to as 'secondary copper production'. Secondary producers use processes similar to those employed for primary production. The ICSG estimates that in 2008, at the producers and manufacturers level, secondary copper refined production reached around 15% of total refined copper production in the world. In 2008, 40 % of all the copper used in the EU-27 came from recycling, compared with 38% in 2007 **[8].**

2.1.2 Uses of Copper

Copper is a kind of non-ferrous metal which has long been closely connected to human beings. Not only are there abundant resources in nature, but copper also possesses excellent properties. Therefore, it is widely used in electrical power, electronics, energy, petrochemicals, transportation, machinery, metallurgy, light and other new industries and some high-tech fields **[6].**

Figures II.1: copper **[6]**

a) Applications in electrical power industry

Electric power transmission, such as wire and cable, transformers, switches, plug components and connectors, etc.; motor manufacturing, for instance as a stator, rotor, shaft head and hollow wire, etc.; communication cables and residential electrical circuits also need to use a large quantity of copper wires.

b) Applications in electronics industry

Copper is used in vacuum electronic devices such as high frequency and ultra-high frequency tubes, crossing the catheter, magnetron, etc. Copper printed circuits require a lot of copper foil and copper base brazing material. In integrated circuits, copper replaces aluminum in silicon chips for interconnection and lead frames.

c) Applications in energy and petrochemical industries

Main condenser tubes and plates are made from brass, bronze and cupronickel in coal-fired power stations within the energy industry. Solar heaters are also often made of copper tube. Different kinds of containers for holding corrosive mediums, pipe systems, filters, pumps and valves, all sorts of evaporators and condensers, while heat exchangers are made from copper and copper alloy in the petrochemical industry. For its corrosion resistance and as soluble copper ions in water it has an antiseptic effect which could protect marine organisms from being polluted; copper and its alloys have been widely used in desalinators and offshore drilling platforms and other undersea installations.

d) Applications in transportation industry

Copper alloy is used in the shipping industry — including aluminum bronze, manganese bronze, aluminum, brass, gun metal (bronze), tin, zinc, copper and nickel copper alloy (Monel), which are all standard materials in shipbuilding. Copper and copper alloy in warships and commercial ships are used commonly to make aluminum bronze propellers, bolts, rivets, condenser pipes, copper coated paint, etc. Copper and copper alloy in the automotive industry are mainly used for radiators, braking systems, hydraulic equipment, gears, bearings, brake linings, power distribution and power systems, gaskets and all kinds of joints, fittings and accessories, etc. On trains, the motors, rectifiers and controls, brakes, electrics and signal systems also rely on copper and its alloys. In addition, railway electrification is a big source of demand for copper and its alloys. The wiring, hydraulic pneumatics and cooling systems of planes all need to use copper. Bearing retainers and gear bearings utilize aluminum bronze pipe, and navigation instruments are made from diamagnetic copper alloy.

e) Applications in mechanism and metallurgy industries

All kinds of transmission parts and fixed parts, such as cylinder liners, gears, fittings, fasteners, twisting, etc., need to use copper or copper alloy for antifriction and lubrication. Also a key part of metallurgical equipment in continuous casting technology – crystallizer is mostly made of chromium copper and silver copper or other copper alloys, which have high strength and conductivity. Besides that, electric vacuum arc furnaces in metallurgy and electro slag furnace water-cooled crucibles are made of copper pipe, while all kinds of induction coil are made of copper or copper winding. Alloy additive copper is an important additive element in steel and aluminum alloy. By adding a small amount of copper to low alloy structural steel, the hardness of steel and its corrosion resistance in air and water can be improved. The addition of copper in corrosion resistant cast iron and stainless steel means their corrosion resistance can be further improved.

f) Applications in light industry

Heat exchangers in air conditioners, clock movements, gridding cloth of paper machines, copper plate printing, fermentation tank lining, distillation pots and architectural decoration components, etc. are all manufactured by using copper and its alloys.

g) Applications in new industries and high-tech fields

It uses such as coating of superconducting alloys, containers and pipelines of cryogenic medium, cooling linings of rocket engines and magnet windings in high-energy accelerators, etc… **[6].**

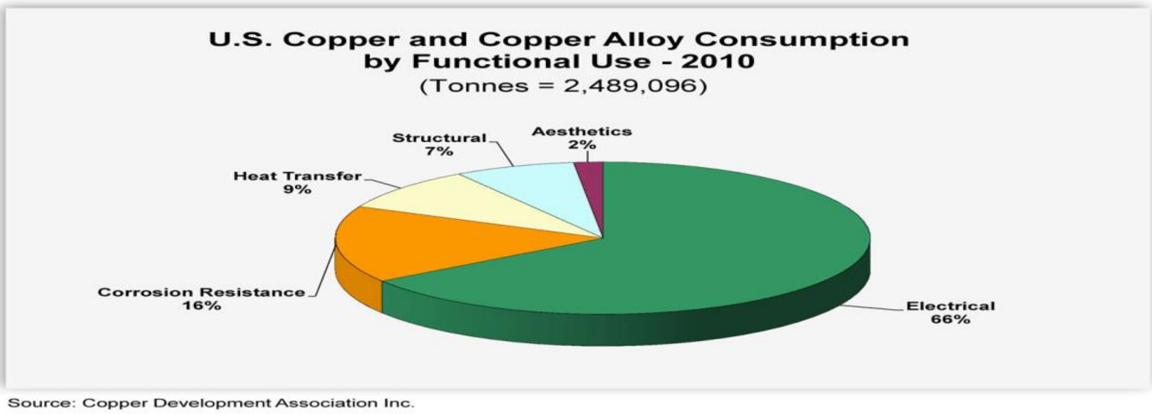


Figure II.2: U.S. Copper and Copper Alloy Consumption by Functional Use-2010-Copper Source: copper development Association Inc. **[6].**

2.1.3 Copper production

Copper results from two sources:

1. Primary production: extraction and processing (refining) of the raw mined material

2. Secondary production, originating from:

- Direct melt of ‘new scrap’ (waste resulting from the manufacturing process)
- Recycling of end-of-life products, using 'old scrap'.

Primary copper production can rely on two processes.

The first process is based on the extraction of copper-bearing ores. After the ore has been mined, it is crushed and ground followed by a concentration by flotation. The obtained copper concentrates typically contain around 30% copper. In the subsequent smelting process, copper is transformed into a 'matte' containing 50-70% copper. The molten matte is processed in a converter resulting in a blister copper of 98.5-99.5% copper content. In the next step, the blister copper is fire refined in the traditional process route, or, increasingly, re-melted and cast into anodes for electro-refining. The output of electro-refining is refined copper cathodes, assaying over 99.99% of copper.

Another important source of raw material is copper scrap. This scrap from either metals discarded in semis fabrication, finished product manufacturing processes ('new scrap') or obsolete end-of-life products ('old scrap'). Upon utilization of scrap, the refining process is referred to as 'secondary copper production'. Secondary producers use processes similar to those employed for primary production. The ICSG estimates that in 2008, at the producers and manufacturers level, secondary copper refined production reached around 15% of total refined copper production in the world. In 2008, 40 % of all the copper used in the EU-27 came from recycling, compared with 38% in 2007 **[8].**

3. Copper Recycling

In many “developed” countries, much of the copper contained in waste is either exported or lost even though a large proportion of the copper in question is not degraded during its use and could, therefore, be recycled. It is estimated that 85% of the copper in circulation is recoverable and that its average utilization time is 30 years (ranging from a few years in electronics applications to over 100 years in the construction industry). Recycled copper is either refined or reused directly (in the case of electric cables, certain alloys, and new manufacturing scrap). The problem of copper recycling is an important issue as copper ranks third by mass in the metals used in the world, after iron and aluminum, and has a wide range of applications. Moreover, pure copper is 100% recyclable indefinitely without any alteration or property loss.

In 2008, for a worldwide annual consumption of 24 Mt of refined copper, 6 Mt came from copper recycled by simple melting and2.7 Mt from copper waste that had been refined. In other words, the total percentage of recycled copper in worldwide consumption was 36.2%. The proportion of recycled copper was 41.4% in Europe,33.5% in Asia and 29.5% in North America

From a technological point of view, many different processes have been developed for recycling metals with fairly efficient technologies. According to the Bureau of International Recycling (BIR,2013), copper recycling reduces energy expenditure by 85% and reduces greenhouse gas emissions by 65% in comparison with primary copper production. Various works are in progress to improve recycling efficiency in specific processing systems, notably concerning WEEE, the category of waste that contains the largest fraction of copper scrap**[3].**

3.1 The Environmental Importance of Copper Recycling

Copper is an essential trace element that is necessary for plant and animal health. Moderate excess exposure to copper is not associated with health risks. As with other metals, there are significant environmental benefits to the recycling of copper. These include solid waste diversion, reduced energy requirements for processing, and natural resource conservation. For example, the energy requirements of recycled copper are as much as 85 to 90% less than the processing of new copper from virgin ore.

In terms of conservation, copper is a non-renewable resource, although only 12% of known reserves have been consumed. Known U.S. reserves of copper are thought to total 1.6 billion metric tons, with production concentrated in Arizona, Utah, New Mexico, Nevada, and Montana. About 99% of domestic production is generated from 20 mines. An emerging environmental challenge for copper is its use in the ever-increasing production of electrical products that still experience low recycling rates. This trend is changing for the better, however, through electronics recycling initiatives **[5].**

3.2 The Economic Importance of Copper Recycling

Copper recycling is much less expensive than new copper extraction and purification. This means that copper recycling helps keep the cost of copper products low and affordable for use in plumbing, electrical cables, etc.

For example, the United States is one of the leading copper-producing countries. The United States produces about 8 percent of the world's copper, and about half of that production comes from recycled copper materials. In 2010, the scrap metal industry in the United States recycled 1.8 million tons of copper for domestic use and export.

Copper recycling enables the United States to remain self-sufficient and positions it as one of the world's largest copper producers **[26].**

3.3 Where to Find Copper for Recycling

For the scrap metal collector, an important source of scrap is an electrical cable, copper flashing, old radiators, and plumbing work. Copper from buildings is crucial, and content is estimated below **[5]**:

1. In houses (estimates for a 2,100 square foot residence)

- 195 pounds - building wire
- 151 pounds - plumbing tube, fittings, valves
- 24 pounds - plumbers' brass goods
- 47 pounds - built-in appliances
- 12 pounds - builder's hardware
- 10 pounds - other wire and tube

2. In an apartment of 1,000 square feet

- 125 pounds - building wire
- 82 pounds - plumbing tube, fittings, valves
- 20 pounds - plumbers' brass goods
- 38 pounds - built-in appliances
- 6 pounds - builder's hardware
- 7 pounds - other wire and tube

3. In residential appliances:

- 52 pounds - unitary air conditioner
- 48 pounds - unitary heat pump
- 5.0 pounds - dishwasher
- 4.8 pounds - refrigerator/freezer
- 4.4 pounds - clothes washer
- 2.7 pounds - dehumidifier
- 2.3 pounds - disposer
- 2.0 pounds - clothes dryer
- 1.3 pounds – range

3.4 Copper scrap processing

Secondary copper recovery is divided into 4 separate operations: scrap pretreatment, smelting, alloying, and casting. Pretreatment includes the cleaning and consolidation of scrap in preparation for smelting. Smelting consists of heating and treating the scrap for separation and purification of specific metals. Alloying involves the addition of 1 or more other metals to copper to obtain desirable qualities characteristic of the combination of metals. The major secondary copper smelting operations are shown in Figure 10; brass and bronze alloying operations are shown in **[13].**

3.4.1 Pretreatment

Scrap processing is achieved through manual, mechanical, Pyrometallurgical hydrometallurgical methods **[8].**

- Manual and mechanical methods include sorting, shredding and magnetic separation. The scrap can then be pressed into briquettes by a hydraulic press.
- Pyrometallurgical pretreatment includes sweating, burning insulation from copper wire and drying in rotary kilns to volatize oil and other organic compounds.
- Hydrometallurgical pretreatment is mainly related to the low quality of residues and includes floating (if slag contains over 10% copper) and leaching to recover copper from slag.

Copper scrap treatment depends on its purity (Figure II.5). The lowest grade scrap is smelted and refined like concentrate in a primary or secondary smelter/refinery. Higher grade scrap is fire refined, then electro refined. The highest grade scrap is often melted and cast without refining **[8].**

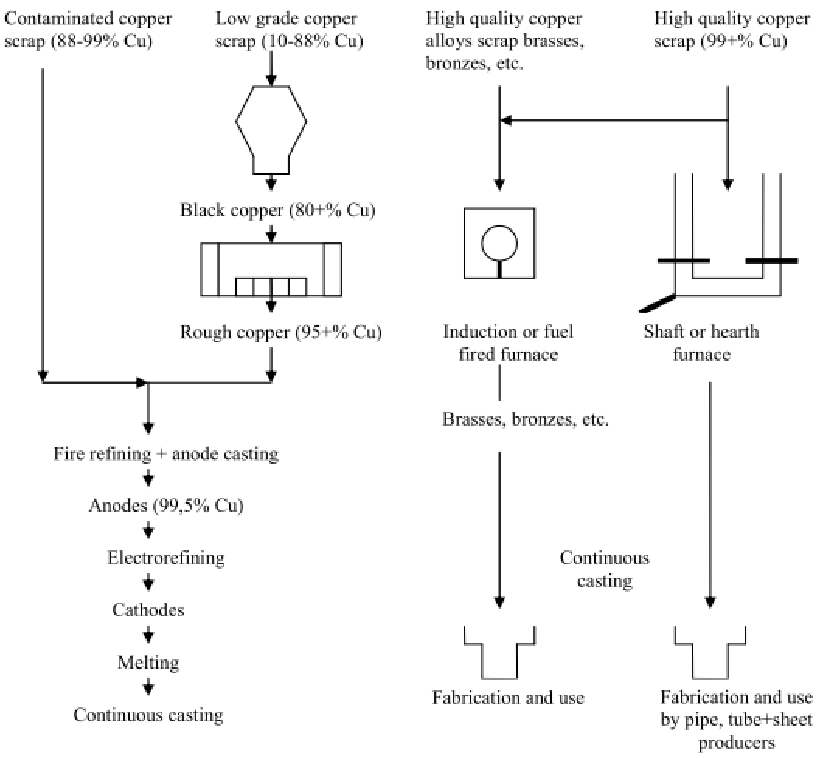


Figure II.5: Flow sheet of processes for the recovery of copper and copper alloys from scrap **[8].**

3.4.2 Smelting

Smelting of low-grade copper scrap begins with melting in either a blast or a rotary furnace, resulting in slag and impure copper. If a blast furnace is used, this copper is charged to a converter, where the purity is increased to about 80 to 90 percent, and then to a reverberatory furnace, where copper of about 99 percent purity is achieved. In these fire-refining furnaces, flux is added to the copper and air is blown upward through the mixture to oxidize impurities. These impurities are then removed as slag. Then, by reducing the furnace atmosphere, cuprous oxide (CuO) is converted to copper.

Fire-refined copper is cast into anodes, which are used during electrolysis. The anodes are submerged in a sulfuric acid solution containing copper sulfate. As copper is dissolved from the anodes, it deposits on the cathode. Then the cathode copper, which is as much as 99.99 percent pure, is extracted and recast.

The blast furnace and converter may be omitted from the process if average copper content of the scrap being used is greater than about 90 percent. The process involves the use of a patented top-blown rotary converter in lieu of the converting, and reverberatory furnaces and the electrolytic refining process. This facility begins with low-grade copper scrap and conducts its entire refining operation in a single vessel **[13].**

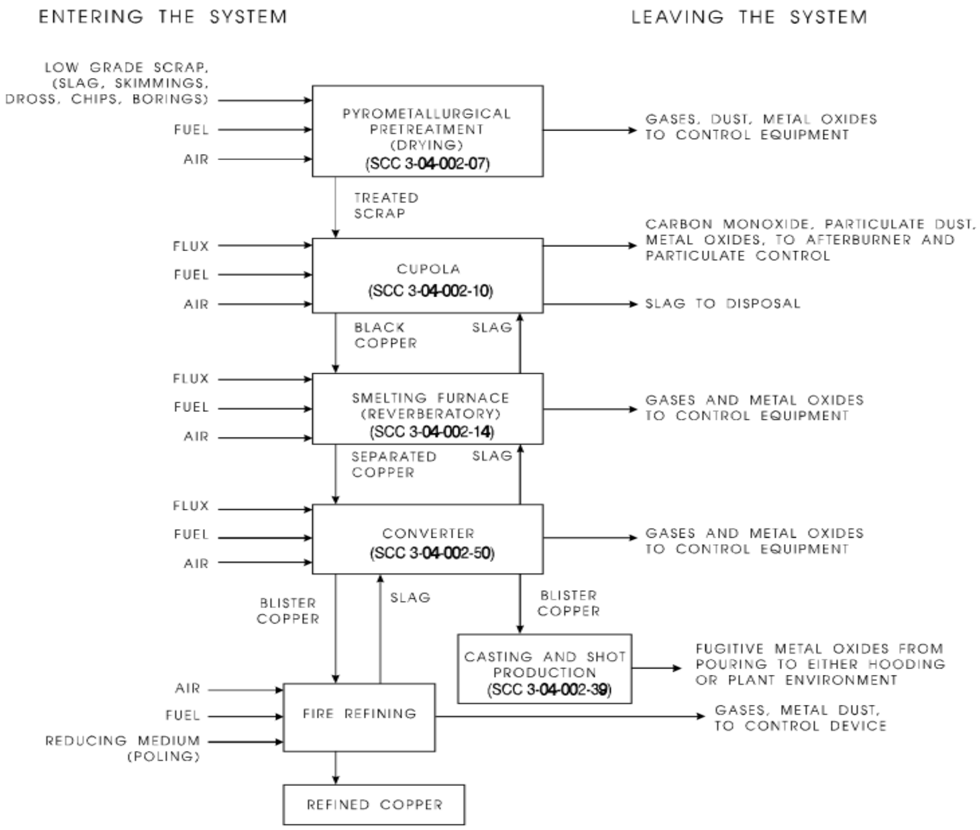


Figure II.6: Low-grade copper recovery **[13].**

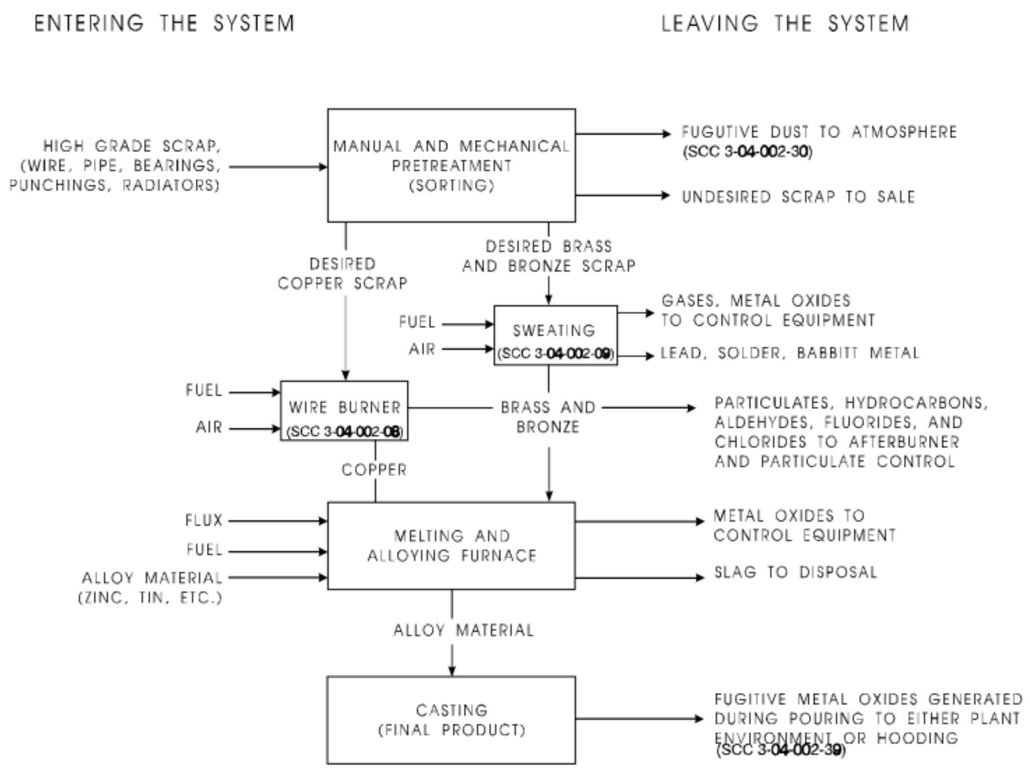


Figure II.7: High-grade brass and bronze alloying **[13].**

3.4.3 Alloying

In alloying, copper-containing scrap is charged to a melting furnace along with 1 or more other metals such as tin, zinc, silver, lead, aluminum, or nickel. Fluxes are added to remove impurities and to protect the melt against oxidation by air. Air or pure oxygen may be blown through the melt to adjust the composition by oxidizing excess zinc. The alloying process is, to some extent, mutually exclusive of the smelting and refining processes described above that lead to relatively pure copper **[13].**

3.4.4 Casting

The final recovery process step is the casting of alloyed or refined metal products. The molten metal is poured into molds from ladles or small pots serving as surge hoppers and flow regulators. The resulting products include shot, wire bar, anodes, cathodes, ingots, or other cast shapes **[13].**

3.5 The Advantages of Recycling Copper

Copper is a 100 percent recyclable material. According to the Copper Development Association, copper's recycling rate is higher than that of any other engineering metal. Every year in the United States, nearly as much copper is recycled as is mined. Excluding wire production, almost 75 percent of U.S. copper used comes from recycled copper scrap. There are so many advantages to recycling copper that the value of scrap is approximately 85 to 95 percent the price of newly mined ore [15].

a. The Decline of Mining

Recycling copper extends its useful life, so much so that, 75% of copper produced since 1900 is still in use. Because recycled copper retains it electrical conductivity, it not only helps increase energy efficiency in many power systems but the copper recycling process consumes up to 85% less energy than its primary production, representing an annual savings of 40 million tons of CO2. In many renewable energy systems, 12-times more copper is used than in traditional systems to ensure efficiency **[30].**

b. Copper Refining

The refining process for copper, releases toxic gases and dust into the air, however, recycling reduces the emissions related to the mining and smelting. According to KME, the Bureau of International Recycling reports that recycling copper saves 85 percent of the energy needed to produce new copper. The amount of solid waste left over from the smelting process is also eliminated, reducing a need for disposal **[15].**

c. Landfills at Capacity

Copper and copper alloy objects which are not recycled might otherwise be dumped in holes in the ground - this is called landfill. These holes are rapidly being filled up and, as they become scarcer, landfill becomes a very expensive option for waste disposal (of any material).**[27].**

d. Mining Waste and Energy

In order to extract copper from copper ore, the energy required is approximately 100 GJ/ton. Recycling copper uses much less energy, about 10 GJ/ton; that's only 10% of the energy needed for extraction. This energy saving leads to the conservation of valuable reserves of oil, gas or coal and reduces the amount of CO_2 released into the atmosphere **[28].**

e. Gas Emissions

Because recycling copper requires less energy than extracting copper from ore, there are fewer gas emissions into the atmosphere, and recycling allows for conservation of valuable resources like coal and oil. Copper alloys might release fumes when melted. Beryllium, for example, is sometimes used in alloys with copper; while beryllium is not dangerous in its solid state, its gaseous state is a known health hazard. Fume extraction equipment can reduce the amount of hazardous gases entering the atmosphere **[27].**

f. Conservation of Copper

The preservation of copper ore is of paramount importance. Although we still have a lot of reserves of it, copper ore is a finite resource and once it is gone, it is gone. We need to recycle as much copper as possible just because we have enough copper already in use to meet current demand **[29].**

g. Landfill Concerns

Without recycling, valuable copper scrap would end up in landfills, which are becoming too full to accommodate more waste. The demand for space in landfills is high, making the cost of dumping waste very expensive. Additionally, buried metals like copper could contribute to environmental harm, including contamination of ground water resources. Recycling copper keeps it from ending up in landfills and causing environmental damage **[27].**

3.6. The Disadvantages of Recycling Copper

Copper is considered a precious metal, most often utilised in electrical components because of its ability to conduct electricity safely and efficiently. Copper's importance in electrical components has increased its value in the marketplace and led to widespread recycling of the metal.

Although recycling limits waste and dependency on copper mines, the task of recycling the metal has a few disadvantages.

a. Difficulty Separating Copper

Copper can be a difficult item to recycle because it needs to be purified of any of metals or contaminants to be used again in electrical components. Pure copper is essential the maintaining the conductivity of the metal. Most copper wiring is used for small electrical wiring and needs to be almost pure, without surface flaws, to prevent breaks during rod production. Recycled copper needs to be uncontaminated to ensure that it can be reused in new electronics. This is a major disadvantage for copper recycling; the process of separating the copper from old electrical components is difficult or not always possible.

b. Expensive

A disadvantage to recycling copper is that unless you have access to your own scrap metal, it can be expensive to buy it from other sources. According to School Science, scrap copper retains around 90 per cent of the cost of original copper, meaning it is almost as expensive to purchase scrap copper as it is new copper. Companies that are looking for sources of recyclable copper are still going to have to pay quite a bit of money for the scrap. This includes only the cost of purchasing the metal and does not include the cost associated with melting down and recycling the copper.

c. Limited Sources

There are two primary areas that scrap metal is drawn from. One is "old scrap" from those who collect and resell old copper. The other is "new scrap," which is drawn from offcuts and scrapings from copper factories. The factories provide a reliable source of copper shavings and offcuts, which are predictably sold back to copper recyclers. The disadvantage comes with the public who provide an unreliable source of the used copper, which is often gathered from old buildings, old copper piping and disused electrical cables. It can be difficult to come across used copper products for recycling because this major source fluctuates regularly **[14].**

Conclusion

According to what we mentioned, we can conclude from this chapter the importance and benefit of copper recycling while preserving its primary properties and achieving important economic goals thanks to it, of course, without forgetting the positive impact on the environmental side. Also, by reviewing the literature, we see that there are several ways to recycle copper; these will serve-us a guide for the realization of our experimental work and able us to achieve the same gains mentioned previously.

CHAPITRE III: Experimental Procedure and Results

Section 1: Materials and Experimental Methods

Introduction

In this chapter we will present the experimental approach pursued for cooper waste recycling resulting from the scrap wire. We will describe the "Oxygen Free" smelting process used for waste recycling. Moreover, we will present the studied substantial namely: the sampling areas, the preparation operations of the collected waste. Also, we will present the main points from the obtained results and the associated interpretations.

We mention that in view of all the analysis and characterization techniques needed to be performed in this study, only optical metallography and chemical analysis were carried out. The others have not been carried out due to the unavailability of equipment.

1. Studied Material

The materials explored in this study are the two types of copper waste and copper cathode. The cathode is known as primary raw material input for the production of copper rod used for wires and cable industry. We refer the two types of copper waste as W1 and W2 and the cathode as C.



Figures III.1: copper waste 1 **(W1)** **Figures III.2:** Copper cathode **(C)** **Figures III.3:** copper waste 2 **(W2)**

2. The chemical composition of the studied materials:

The chemical composition of the materials studied is illustrated by the following tables. Note that we only have that of the cathode and that of the waste w2.

Table III.1: Chemical compositions of cathode C.

	Zn	Pb	Sn	P	Mn	Fe	Ni	Si
%	0.0648	0.0021	<0.00050	0.00051	<0.00020	0.0020	<0.00050	<0.00060
	Cr	Te	As	Sb	Bi	Ag	Co	Al
%	<0.00020	0.0018	<0.00040	< 0.0035	<0.00060	<0.00030	< 0.0015	0.0121
	S	Be	Zr	Au	B	C	Ti	Se
%	<0.00020	<0.00010	<0.00020	<0.00050	<0.00020	0.0121	<0.00020	<0.00080
	Nb	Pt	Cu					
%	<0.0010	<0.0020	99.9					

Table III.2: Chemical compositions of the waste 2 **(W2)**.

	Zn	Pb	Sn	P	Mn	Fe	Ni	Si
%	0.123	0.319	0.234	<0.00050	<0.00020	0.0837	0.0696	<0.00060
	Cr	Te	As	Sb	Bi	Ag	Co	Al
%	<0.00020	<0.00030	0.00049	<0.0035	<0.00060	0.0119	<0.0015	0.0099
	S	Be	Zr	Au	B	C	Ti	Se
%	0.0410	<0.00010	<0.00020	<0.00050	<0.00020	0.0259	<0.00020	<0.00080
	Nb	Pt	Cu					
%	<0.0010	<0.0020	99.1					

3. Preparation of samples

The samples prepared for the fusion are method C, the two wastes W1, W2 and their mixtures two by two. These mixtures are made in fixed proportions where the quantity of each component is weighed with a type microbalance RADWAG PS 600R2. And to get an idea of the evolution of chemical elements after fusion, the mixtures investigated are as follows:

The materials: (100% of C), (100% of W1) and (W2 100% of W2)

The Mixtures: (40% of C with 60% of W1), (40% of C with 60% of W2), (60% of W1with 40% of W2) and (40% of W1with 60% of W2)

For example: if we take a mixture of 30 grs, the quantity of the elements will be: 40% wt. (12g) of one and 60% wt. (18g).

W2 40% **W1 60%** **W2 60%** **W1 40%**

C 60% **C 40%**

Figures III.4: The weighing

3.1 Smelting Process

From the copper data sheet; the measured melting temperature is 1084.62°C. given the available means, we considered it appropriate to work at a temperature of more than 1100 ° C at different times.

The experimental parameters that can be dealt with are the temperature, the retention time and the type of cooling

3.1.1 Smelting process equipment

The copper smelting process requires three basics equipment:

- **Refractory crucible:** Where the sample to be melted is placed and then entered into the melting furnace, which is highly heat-resistant.

Figures III.5: Shape of Refractory used in smelting

- **Melting furnace:** It is a special furnace used to melt metals that can reach a temperature of up to 1500 C° which is more than enough to smelt copper, who’s the melting point is 1085 C°.

Figures III.6: a melting furnace

- **Charcoal**: It is placed above the sample inside the refractory crucible to prevent oxidation of the copper. Indeed, the oxygen of the ambient air will not be able to reach the metal (the copper) since it will be consumed by the carbon of the charcoal.

This is the best cheaper method of copper smelting, but unfortunately there were some problems that prevented us from doing it, because of the lack of material.

Figures III.7: a coal

We had to use heat treatment furnace its maximum temperature is 1200 C°,

Figures III.8: heat treatment furnace

we did the operation but we got a bad result, the smelting process did not come as perfect way because we found that some foreign reactions occur at the level of the furnace that we don't know about it yet.

Figures III.9: Some results obtained from the use of a heat treatment furnace

After some waiting time, we managed to get a melting furnace, the maximum temperature that can be reached is 1500C.But unfortunately this temperature could not be reached due to a problem encountered in the programming software. And even the temperature required for copper smelting could not be achieved with this furnace.

Figures III.10: Melting Furnace

Figures III.11: The result obtained with Melting furnace

In order to be able to complete our work, we resorted to fusion by high intensity electric current. This method requires certain equipment such as:

- **Inverter welding machine:** It is used to provide the electrical energy necessary for fusion; it is used as an amplifier of electrical intensity.

Figures III.12: Inverter welding machine type SALI MMA-250

- **Graphite electrodes**: Graphite is known for its very high electrical resistivity which confers it to be used as a plasma electrode or as an electrical resistance with Joule effect. The graphite electrodes used to melt metal component are small cylindrical graphite bars that have been taken out of batteries, as can be seen in the figure below. The graphite electrode by touching the surface of the sample causes strong heat transfer and some plasma reaction to the metal which produces its melt.

Figure III.13: Graphite electrode extracted from batteries

- **Refractor brick:** It is a brick of refractory material in which we made a hole to use it as a melting refractory crucible.

- **Charcoal:** These are black embers of charcoal of different sizes, like it can be seen in the figure below. It is used as a preventive barrier against oxygen from the ambient air in order to prevent it from reaching the sample.

Figure III.14: Photo of the charcoal used

3.1.2 Protective equipment

Due to the fact that we dealt with many risks that threaten our health during the operation, we needed some equipment for personal protection like:

- **Leather apron:** A leather apron is a garment that generally covers the front of the body, and worn primarily for protection of one's body and clothing from sparks, sharp objects, heat and possible splashes of molten metal.
- **Welding goggles and Face Mask:** This equipment is intended to protect the eye and face from high heat and light radiations, such as intense ultraviolet light from an electric arc, and also from sparks or debris.
- **Gloves:** The gloves are personal protective equipment (PPE) that protects the hands. These gloves allow protecting the operator from electrical shock, extreme heat, and ultraviolet and infrared radiation, and also and provide better grip for holding equipment.
- **Mask:** We use it to protect ourselves against inhalation of smoke and gases emitted during the procedure.

Figure III.15: Protective equipments

After assembling the equipment and donning the protective gear, we were finally able to perform the merger as shown in the figures below.

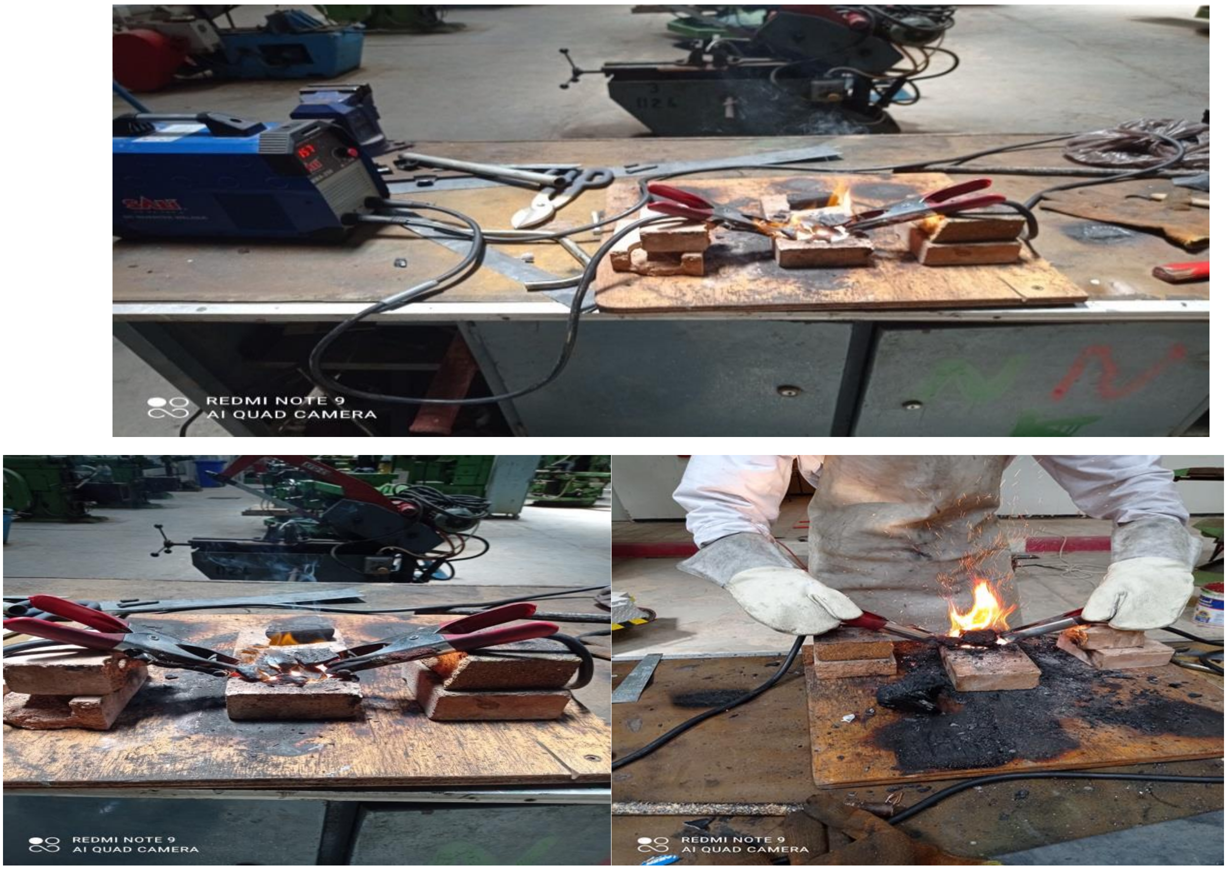
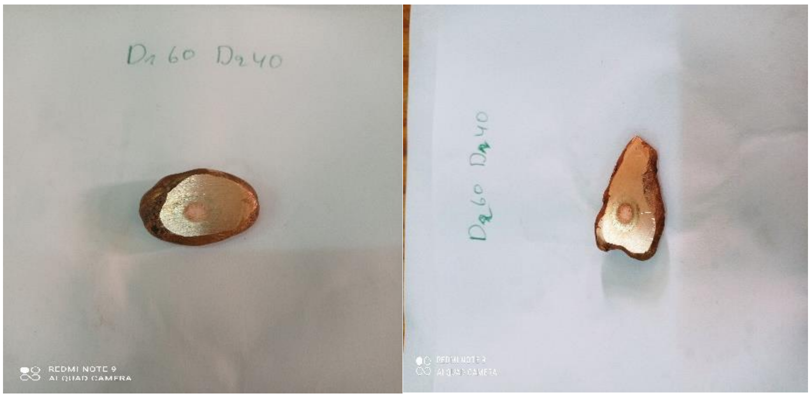


Figure III.16: Smelting process with a High intensity electric current

C 100% **W2 100%** **W1 100%**



W1 60% W2 40% **W2 40% W1 60%**

C 40% D2 60% **C 40% D1 60%**

Figures III.17: Forms of samples obtained after the melting process and preparation the surface

3.2 Sample’s preparation for analysis

In view of the analyzes that we carried out in this study, namely: chemical composition and optical metallography, the preparation operations only requires mechanical polishing and chemical attack.

3.2.1 Mechanical polishing

After cutting, we obtain a very rigorous surface, we move on to mechanical polishing with abrasive papers which aim to reduce the roughness and improve the appearance. The different grain sizes of the papers (from 120 to 1200) are used depending on the desired surface.Water is used as a lubricant during polishing, except that for the final polishing with felt paper oil and diamond paste with 1 µm granulometry was applied.

Figure III.18: Mechanical polishing process

3.2.2 Metallurgical attacks

Chemical etching is a chemical process that follows mechanical polishing. The chemical attack reveals the contrast of the grain boundaries and the different phases present in the sample. Each substance has a specific reagent linked to the purity and the elements present. For copper, in general, we use nitric acid to reveal the microstructure.

4. Characterization Techniques

Chemicals analysis

The analysis of the chemical composition was carried out using the spectro-Max apparatus illustrated in the figure III.19. This analyzer has a good precision of up to 10^{-6}.

Figure III.19: Photo of the Spectro-MAXx Analysis

Optical microscopy

For a primary understanding of the state of sample fusion that have been carried out in microscopic observation, because we can observe and determine, the evolution of the grains (size, shape, porosity….), The distribution of precipitates and other elements present, grain boundary movement, twinning, and many other structural features. To do this, we use a metallographic mid-ceroscopy of the OPTIKA type fitted with a digital camera which allowed us to take micrographs of the structure of the samples studied for magnifications of up to 1000 times.

Figures III.20: Photo of the Optical microscopy type OPTIKA

Section 2: Results and discussions

Introduction

In this part, we will present the results obtained after the fusion technique by high intensity electric current. We first present the results of the chemical composition analysis, then the microstructures of some elaborate fusion.

1. Chemical Analysis Results

a. Copper cathode (100% of C)

Table III.3: Chemical composition of the melted cathode (100% of C)

	Zn	Pb	Sn	P	Mn	Fe	Ni	Si
%	0.0138	<0.0010	<0.00050	<0.00050	<0.00020	0.0031	<0.00050	<0.00060
	Cr	Te	As	Sb	Bi	Ag	Co	Al
%	<0.00020	<0.00030	<0.00040	< 0.0035	<0.00060	<0.00030	< 0.0015	0.0031
	S	Be	Zr	Au	B	C	Ti	Se
%	0.0073	0.0193	<0.00020	<0.00050	<0.00020	> 0.0732	<0.00020	<0.00080
	Nb	Pt	Cu					
%	< 0.0010	< 0.0020	99.9					

Through the results of the chemical analysis of the copper cathode after the smelting process and comparing them with the preliminary chemical composition **(table III.1)**, we found that the amount of copper **Cu** was not affected at all, as its percentage still **99.9%** in the melted state. The same is true for the rest of the elements, as they did not have any change except with the intention of carbon **C**, which needs to be more reviewed where it recorded a noticeable rise where arriving up to> 0.0732%. This is certainly due to the use of charcoal in the process.

b. Copper waste 1 (100% of W1)

Table III.4: Chemical composition of the melted copper waste 1 (100% of W1)

	Zn	Pb	Sn	P	Mn	Fe	Ni	Si
%	0.0159	<0.0010	<0.00050	<0.00050	<0.00020	0.0072	<0.00050	<0.00060
	Cr	Te	As	Sb	Bi	Ag	Co	Al
%	<0.00020	0.0010	<0.00040	< 0.0035	<0.00060	<0.00030	< 0.0015	0.0215
	S	Be	Zr	Au	B	C	Ti	Se
%	0.0017	<0.00010	<0.00020	<0.00050	<0.00020	> 0.0732	<0.00020	<0.00080
	Nb	Pt	Cu					
%	< 0.0010	< 0.0020	99.9					

After the process of smelting waste copper 1 **(100% of W1)** and despite the absence of our initial chemical composition, we obtained the results of its chemical analysis, where we recorded a very high value of the percentage of copper Cu that reached to **99.9%**. As in the previous case, we note the increase in the content of the Carbone after melting this waste.

c. Copper waste 2 (100% of W2)

Table III.5: Chemical analysis of the melted copper waste 2 sample (100% of W2)

	Zn	Pb	Sn	P	Mn	Fe	Ni	Si
%	0.0376	0.227	0.0800	<0.00050	< 0.00020	0.0266	0.0199	< 0.00060
	Cr	Te	As	Sb	Bi	Ag	Co	Al
%	<0.00020	0.00072	0.00077	<0.0035	<0.00060	0.0095	<0.0015	<0.00050
	S	Be	Zr	Au	B	C	Ti	Se
%	0.0136	<0.00010	<0.00020	<0.00050	<0.00020	>0.0732	<0.00020	<0.00080
	Nb	Pt	Cu					
%	< 0.0010	< 0.0020	99.5					

Comparing the results obtained through chemical analysis for copper waste 2 (**W2 100%)** with its own primary chemical composition (table III.2) we notice an increase in the proportion of copper **Cu** inside the melted sample; where it was **99.1%** and became **99.5%.** Logically, it is unreasonable to record this increase, but after examining the results in the table, we noticed a severe decrease in the proportion of some elements like **S, Zn, Pb, Sn,** after the smelting process from. For S and Zn, vaporization may be the cause of the decrease in their quantity in the molten sample, since their vaporization points (445 ° C and 907 ° C respectively) are located very low compared to the point of copper melting (1084 ° C). However, heavy elements such as Pb and Sn a possible settling due to their density could lead to lower their content after melting. But it remains one possibility among many that requires further scrutiny.

d. Mixture (40% of C+ 60% of W1) and (40% of C + 60% of W2)

The copper cathode (C) was mixed with various copper waste (W1, W2) in the rate of (40% .60%) in sequence the following results were obtained

Table III.6: Chemical analysis of the Mixture (40% of C+ 60% of W1) sample

	Zn	Pb	Sn	P	Mn	Fe	Ni	Si
%	0.0163	< 0.0010	<0.00050	<0.00050	<0.00020	0.0030	<0.00050	<0.00060
	Cr	Te	As	Sb	Bi	Ag	Co	Al
%	<0.00020	<0.00030	<0.00040	< 0.0035	<0.00060	<0.00030	< 0.0015	<0.00050
	S	Be	Zr	Au	B	C	Ti	Se
%	0.0017	<0.00010	<0.00020	<0.00050	<0.00020	>0.0732	<0.00020	<0.00080

	Nb	Pt	Cu					
%	<0.0010	<0.0020	99.9					

Table III.7 : Chemical analysis of the Mixture (40% of C+ 60% of W2) sample

	Zn	Pb	Sn	P	Mn	Fe	Ni	Si
%	0.0603	?0.0600	0.119	>0.144	<0.00010	>0.276	<0.00020	>0.0720
	Mg	Cr	Te	As	Se	Sb	Cd	Bi
%	0.0050	0.264	0.0027	>0.348	0.0040	>0.312	> 0.0384	0.0206
	Ag	Co	Al	S	Be	Zr	Au	B
%	0.0024	0.0603	>0.0480	>0.0360	0.00078	0.0132	<0.00050	>0.0120
	Ti	Pt	Cu					
%	<0.00020	<0.0020	98.1					

As in the previous case, we note the change in the content of certain elements after melting compared to the reference states of the mixture. Depending on the tables above, we found that the percentage of copper **Cu** in the sample **(C40%, W1 60%)** which is 99.9% is higher than its percentage in the sample **(C40%, W2 60%)** which is 98,1%. We also note the increase in iron and carbon in the mixture (C40%, W1 60%); this negatively affects the electrical resistivity of the wires produced from this mixture. Regarding the increase in carbon content, this can be attributed directly to charcoal dust and ash trapped in the smelting. It should also be pointed out that the absence of carbon in the mixture **(C40%, W2 60%)** is more of an anomaly than a result to be explained.

Through this and by comparing the two samples, we find that the combination between copper cathode C and Copper waste W1 is better than with the copper waste W2.

e. Mixture (60% of W1+40% of W2) and (60% of W2+40% of W1)

The two types of copper waste (W1, W2) were mixed in the rate of (40% and 60%) After carrying out the chemical analysis, the following results were obtained.

Table III.8: Chemical analysis of the melted Mixture sample (60% of W1+40% of W2).

	Zn	Pb	Sn	P	Mn	Fe	Ni	Si
%	0.0292	0.0474	0.0326	0.00081	<0.00020	0.0093	0.0084	0.0153
	Cr	Te	As	Sb	Bi	Ag	Co	Al
%	<0.00020	0.00071	0.00057	< 0.0035	<0.00060	0.00040	< 0.0015	0.0036
	S	Be	Zr	Au	B	C	Ti	Se

%	0.0090	<0.00010	<0.00020	<0.00050	<0.00020	>0.0732	<0.00020	<0.00080
	Nb	Pt	Cu					
%	<0.0010	< 0.0020	99.8					

Table III.8: Chemical analysis of the melted Mixture sample (60% of W2+40% of W1).

	Zn	Pb	Sn	P	Mn	Fe	Ni	Si
%	0.0350	0.214	0.0893	<0.00050	<0.00020	0.0312	0.0404	<0.00060
	Cr	Te	As	Sb	Bi	Ag	Co	Al
%	<0.00020	0.00069	0.00061	< 0.0035	<0.00060	0.0048	<0.0015	<0.00050
	S	Be	Zr	Au	B	C	Ti	Se
%	0.0179	<0.00010	<0.00020	<0.00050	<0.00020	>0.0732	<0.00020	<0.00080
	Nb	Pt	Cu					
%	<0.0010	<0.0020	99.5					

In the case of waste mixtures, we also notice the same behavior of changes in the contents of the elements in one direction or the other (increase and decrease).

By comparing the two samples, we found that the highest percentage of copper Cu is present in the sample (W1 60 % W2 40%) by up to 99.8%. Through this we can say that the combination (W1 60%, W2 40%) is better than (W2 60%, W1 40%) for use in the field of electric cable.

2. Microstructural analysis

After preparing the surface of the samples and placing them under the optical microscope, we obtained the following results, which were all similar for all samples, and to illustrate the results we show two examples of that.

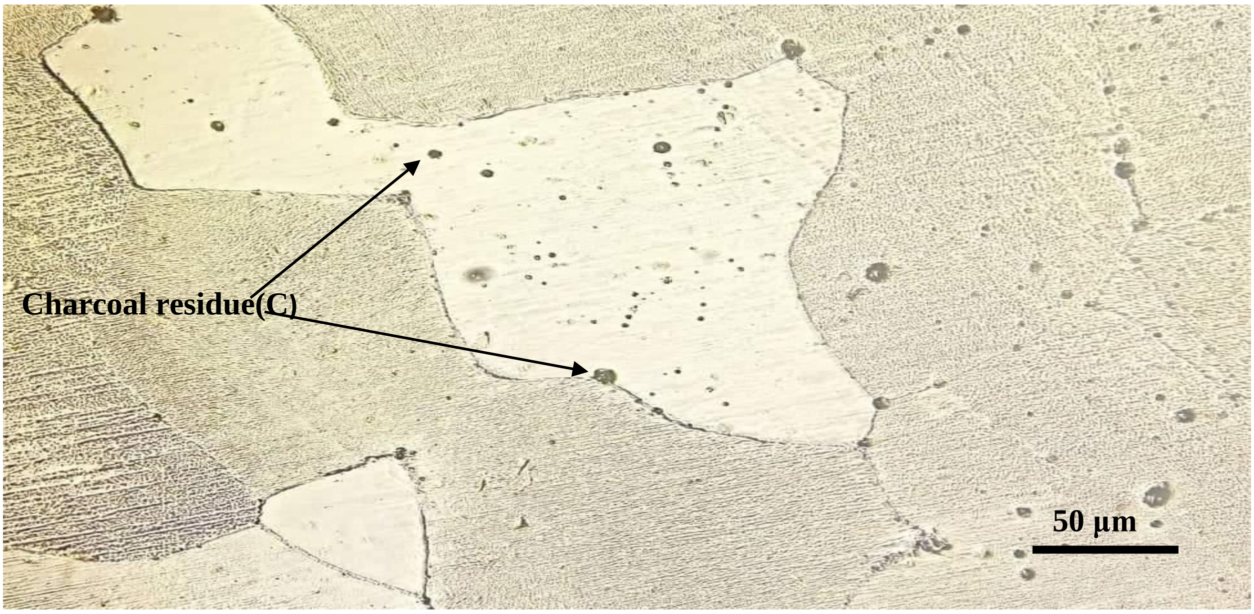


Figure III.21 : The microstructure of the melted sample 100% of C

Figures III.22: The microstructure of the melted sample (C40%, D260%)

From what we have obtained, we can say that we got the normal microstructure of copper after melting with a large grain size. We noticed the presence of charcoal residues (black dots) at the grain boundaries and in the grain matrix. This partly explains the increase in the carbon content appearing in the chemical composition of the samples after melting. This is certainly related to the melting technique used in this work and to the quantities of material remelted. Given the small quantities used (30 gr) and given the very rapid melting of the metallic mixture (a few minutes); dust and charcoal ash present inside the mixture will not have time to float on the surface will remain trapped in the molten mass. However, in the case of melting large quantities as is done in industrial furnaces, this phenomenon does not occur since these light ashes will remain in flotation at the top of the bath.

Conclusion

From the results mentioned in this chapter, we find that the best samples in terms of percentage of copper value are: (C40%, W1 60%) and (W1 60%, W2 40%) at the rate of 99.9% and 99.8% in sequence. However, due to the high cost of the copper cathode, the mixture (W1 60%, W2 40%) will present the right compromise in terms of economy and resources.

As for Copper 2 (W2) waste, it needs more refining before it is used in the manufacturing process.

Regarding the increase in the carbon (C) content observed after melting, we need not worry, because this phenomenon does not occur in industrial production.

GENERAL CONCLUSION

The aim of our study was to recycle copper waste that belong to Biskra Cable Industries Company (ENICAB BISKRA) and to find the perfect combination between the three types of copper (copper cathode C, copper waste1 W1, copper waste 2 W2)

From the results obtained, the following points can be concluded:

- ✓ Re-melting copper and its waste under a charcoal layer gives good results in terms of protection against oxidation.
- ✓ High intensity electric current reflow can be a fast and efficient alternative technique in terms of melting copper and preserving its chemical composition.
- ✓ The mixture suitable for the production of electric wires, in terms of their properties only, is certainly the mixture (40% of C with W1 60% of W1). However in terms of the trade-off between electrical properties and production costs, the mixture of scrap copper (60% of W1with 40% of W2) becomes the most suitable for the company.

In Perspective

To better enrich this subject, additional analyzes and characterizations such as the measurement of the rate of oxygen and measurement of the electrical resistivity are more than desirable.

When it comes to measuring the mechanical properties of the wires, this work is also very interesting, but it is difficult to carry out in the laboratory because it requires assembly and suitable materials.

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ملخص

هذه الدراسة هي إعادة تدوير بعض نفايات النحاس. في هذا العمل استخدمنا تقنية صهر النحاس باستعمال التيار الكهربائي عالي الشدة، التي أثبتت ناجعتها في صهر النحاس. وباستخدام تقنيات التحليل والتوصيف مثل التحليل الكيمائي والمجهر الالكتروني تحصلنا على بغض النتائج الإيجابية. وجدنا أن النوع الأول من نفايات النحاس **(W1)** هو الأكثر ما يجب الاستثمار فيه من أجل تخفيض تكلفة الإنتاج. و مع مزجه مع النوع الثاني من نفايات النحاس **(W2)** بنسب **(W1 40%,W2 60%)** شكل مزيجا فعالا من ناحية خصائصه المطلوبة و من الجهة الاقتصادية. أما بالنسبة إلى النوع الثاني من نفايات النحاس **(W2)**، فهو يحتاج إلى المزيد من التنقية ليستعمل بنسب عالية في إنتاج الأسلاك الكهربائية.

Abstract

This study deals with the recycling of scrap copper. For fusion, we exploited the effect of high-intensity electric current. And for the analysis and characterization we used the optical microscope and spectroscopy. We have found that the first type of copper waste (W1) is the most to invest in, in order to reduce the cost of production. With another waste (W2) they can constitute an effective mixture (W1 100% with W2 60%) in terms of desired properties and in terms of costs. As for the second type of scrap copper (W2), it needs more refining in order to increase its percentage in the electrical wire.

Résumé

Cette étude traite le recyclage des déchets de cuivre. Pour la fusion, nous avons exploité l'effet du courant électrique à haute intensité. Et pour l'analyse et la caractérisation nous avons utilisé le microscope optique et la spectroscopie. Nous avons trouvé que le premier type déchet du cuivre **(W1)** est le plus dans lequel il faut investir afin de réduire le coût de production. Avec un autre déchet **(W2)** ils peuvent constituer un mélange efficace **(40% de W1 et 60% de W2)** en termes de propriétés recherchée et en termes de coûts. Quant au deuxième type de déchets de cuivre **(W2)**, il a besoin de plus de raffinage afin d'augmenter son pourcentage dans le fil électrique.