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RECIKLIRANJE METALA IZ OBNOVLJIVIH IZVORA ENERGIJE U LEGURE KOJE SE KORISTE U ZELENIM ENERGIJAMA

RECYCLING OF METALS FROM RENEWABLE ENERGY SOURCES TO ALLOYS USED IN GREEN ENERGIES

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Reciklaža specifičnog elektronskog otpada iz obnovljivih izvora energije pruža mogućnost da se on integriše sa pirometalurškim proizvodnim procesom različitih vrsta obojenih legura. Jedna vrsta legura se koristi za lemljenje različitih vrsta materijala kao što su bakar i nerđajući čelik i često se koriste za instalacije solarnih kolektora. Ostale dobijene legure pripadaju novom tipu legura srebra otpornih na tamnjenje koje se mogu koristiti u elektronici i raznim aplikacijama obnovljivih izvora energije. Svi reciklirani metali iz procesa se koriste za kasniju ingot metalurgiju i mogu se dalje prerađivati da bi se dobila žica, lim ili drugi odgovarajući oblici (šipke, trake). Ova studija prezentuje proces reciklaže sa tehnološkim parametrima i tokovima materijala. Posebna pažnja u radu je posvećena strukturi odabranih legura. Za karakterizaciju mikrostrukture legura korišćeni su optička mikroskopija, SEM sa EDS-om i XRD metoda.

***Ključne reči**: Recikliranje materijala iz OIE, Legure, Struktura, Integrisani procesi*

Recycling specific e-waste from renewable energy provides an opportunity to integrate it with a pyrometallurgical production process of various types of non-ferrous alloys. One type of alloy is used for brazing of different types of materials like copper and stainless steel and they are often used for solar collector installations. Other obtained alloys belong to the new type of tarnish-resistant silver alloys that can be used in electronics and various renewable applications. All recycled metals from the process are used for the later ingot metallurgy and could be further processed to obtain these alloys' wire, sheet metal, or other suitable shapes (rods, strips). This study presents the recycling process with the technology parameters and material flowsheets. Special attention in the paper is given to the structure of selected alloys. Optical microscopy, SEM with EDS, and the XRD method were used to characterize the microstructure of the alloys.

***Keywords**: Renewable Recycling, Alloys, Structure, Integrated processes*

1 Introduction

The evident impacts of climate change have led to an accelerated transition to renewable energy sources (RES), especially after the Paris Agreement. Technological innovation will enable this energy transition, notably in the field of renewable energy. Not only newly installed renewable energy power capacity be critical, but also the rapidly falling costs and competitiveness of all RES sectors, including lowering the use of critical raw materials (primarily metals) [1]. However, despite predicted improvements, the transition to renewables will require a new system of material use to support its infrastructure, including wind and solar energy, energy storage (batteries, hydrogen), and other supporting technologies [2].

Potential supply bottlenecks that could inhibit the future deployment of renewable energy technologies. At the same time, such concerns are not expressed for the primary bulk materials used for renewables, electrical grid, and other infrastructure, like copper and aluminum [3]. The ultimate primary resources of Cu are not a concern for the scarcity of the resource by 2070, and the primary copper supply would be the main source of the total supply till then. However, some Cu sensitivity analyses have shown that recycling processes may become the main copper source of supply by 2070 [4]. The importance of basic technical metals (Cu, Al, Zn) additionally includes several issues. The related mining and industries account for significant global greenhouse gas (GHG) emissions and other environmental considerations [3]. The gross energy requirement (GER) of copper in 2050 with a 100% renewable energy system is expected to be two to sevenfold larger than in 2013, depending on technological progress, the recycling rate, and the future electricity demand. Nevertheless, even in a high recycling rate scenario, complete prevention of GER cannot be expected, although it can significantly reduce that problem [5]. This is an example that recycling is not a solution to full compensation for missing raw materials and other problems with base metals but is not proof that recycling is necessary since it is required for the sustainable use of copper after 2070 [5].

On the other side, recycling is urgent for most critical metals. About 30 metals, including the rare earth elements (REE), are considered critical for accelerated use of RES with the aim of zero carbon emission till 2050. Depletion horizons for several of them are within that timeframe. The significance of recycling should be outlined because even an increase of 50% in the recycling rate (e.g., from 50% to 75% for silver) would not be enough to extend the depletion period seriously [6].

Wind energy is less demanding than other RE sources in the number of critical metals needed since it uses only a few REEs, significantly only neodymium (Nd) and, in less extent - dysprosium (Dy) [7]; although accompanying technologies (electronics, batteries, etc.) use several different metals with limited reserves. On the contrary, standard photovoltaic cell technologies spend several metalloids (B, Ge, Si, Se, Te) and many metals (over 20), most of which belong to the critical group.

The particular importance of silver and indium as materials for solar photovoltaic panels should be emphasized. Crystalline silicon photovoltaics are still the most used today, with 80% of the global PV markets in 2011, and estimates of the silver content were about 8 g/m^2 at that time, primarily for screen-printed silver paste technology for this type of panels. Simultaneously, the silver content for the concentrated solar power is nearly the same for all such technologies and is about 1 g/m^2 per mirror area, with some differences in efficiency and thus per installed kW [8]. Thin film cells based on amorphous silicon (a-Si) have relatively low efficiency but are also widely used. The front contact is an ITO layer (indium-tin-oxide), typically 60 nm thick This means the need for indium is approximately 0.4 g/m^2 [9].

Most metals used in RES have a recycling rate between 25% and 50% (where silver belongs), but some REE and metals have just 1% or less (e.g., B, Li, Ga, Ge, Se), which is also the rate for indium [10]. This article aims to point out the possibilities of increasing these recycling rates with an emphasis on silver and indium as critical metals for (and because of) renewable energy sources, along with less critical but also important metals - copper and zinc. In addition, it shows the summarized results of laboratory research on recycling these materials from REE and the possibility of an integrated process of obtaining alloys that are used for the same purpose as an added value to the recycling process. The characteristics and structure of these alloys, as significant materials, are especially emphasized.

2 Discussion

2.1 Recycling potential of silver and indium

Silver recycling rate from renewables could reach more than 90% (95% for batteries, which is about double the current rate); however, only about 80% could be achieved from solar PV. It is considered the total recycle rate (RR), including potential maximal collection rate (85%) and technological recovery efficiency (as high as 95%), resulting in about 80.8% [2]. In this case, critical is the collection rate, which should be almost doubled, and even then, not high enough to achieve very high RR (above 90%). Technologies that combine different types of metallurgical processes have been available for more than a decade [11] and are extremely efficient (99% or more). Even simple hydrometallurgical methods can achieve recovery of over 97%, and in combination with the electrometallurgical process, practically analytical recovery (99.98%) [12] in laboratory conditions. These results prove that mean industrial recovery could easily reach assumed earlier (95%). Much lower consumption of Ag is expected with the application of the new PV technologies that should reduce its use fivefold, from 20 kg/MW of installed power to just 4 kg/MW [13].

The main indium source is still from mining. Mining and metallurgy of indium are associated with base metals ores containing zinc and copper (the most significant source); indium is obtained only as a by-product due to its low concentrations. The developed model of indium demand suggests that it will be dominated by CIGS PV technology in the future (2030 and onwards). Quantitative sensitivity analysis indicates that parameters associated with thin-film PV technology advance would be the most influential on indium demand. Recycling indium could have an impact that would not be negligible, but even more important should be the recycling of zinc, which is expected to be constant at the rate of nearly 15%. The higher zinc recycling rates would lower the quantity of In from the primarily (ore) sources [14].

Regarding recycling, even in the EU, 60 of total 68 tons (yearly consumption in 2014) were not recovered, of which almost all recovered In was from production scape (about 6 tons) [15]. Potentially, soldering and brazing alloys of indium could be important raw materials for recycling, but only with selective separation and targeted recycling of those alloys [15, 16]; this is exactly the approach that was applied in the research of the author's team related to the topic of this paper and the central theme of the manuscript [17-20]. Although not directly RES, LED technology has a strong positive influence on lowering global greenhouse gas emissions. LEDs are a potentially valuable source of different critical metals, especially Ag and In. The theoretical potential of materials available for recovery just in LED lamps (not all the LEDs) by waste generated between 2017 and 2030 in EU member states is about 60 tons of Ag and nearly 300 kg of In. Collection and recycling rates are expected to be 80% and 85% between 2017 and 2030 in the EU, leading to a recovery rate of 68% [21]. Although these are relatively high percentages, it leads to 20 tons of silver loss. Regarding the technological possibilities, higher recycling rates could be expected since laboratory studies have shown 85.7-98.6% for indium and 89.3-98% for silver, depending on LED types and treatment; even 99.9% and 99.99%, respectively, in some specific cases [22].

2.2 Recycling indium from Solar PV

Indium recycling from CIGS modules at the industrial level seems less well developed than other critical elements, like Te from CdTe modules. Future recycling rates from end-of-life CIGS modules are still undeterminable. Scarcity of indium after 2030 and especially after 2050 will lead to a drastic increase in price and could intensify indium recycling from this source [23]. Serious and extensive studies from the middle of the second decade of the 21st century had severe objections to the high costs of recycling. CIGS nodule recycling cost estimates for indium gave about 3450 $/kg, and its price then was just 700-800 $/kg. Such disproportion between cost and price suggests that recycling technologies must be more economical to become cost-effective [24]. The only two companies that have established recycling processes for CIGS at the industrial level till now are Umicore (Belgium) and 5N Plus (Canada). However, those technologies are intellectual properties of the companies and without publicly available details [25].

The effective recovery of ITO from functional and EOL perovskite solar cell devices was demonstrated in a recent paper. Authors claimed that the developed technique could be modified to recover valuable components in other thin film solar cells. The proposed method was based on the KOH solution and oriented toward industrial application. It can be considered very simple, fast, and environmentally acceptable [26]. Similarly and even more effective methods could be found in two contemporary studies. Selective alkali leaching was applied to separate indium and gallium effectively in the former and more similar to the previous. The process uses a high concentration of NaOH to leach Ga, whereby In remains in the residue (nearly 97% of it) and is further processed by acid leaching. High purity oxides of these two metals were finally obtained [27]. The latter described the high-yield process of all components from CIGS. The method combines acid leaching with selective solvent extraction, precipitation, and calcination. Firstly, indium was transferred to the organic phase, while Ga was extracted using the same extraction agent under different conditions. Consequently, Cu remained in the aqueous solution with just traces of In and Ga. Finally, all three metals from different solutions, at different pH values, were chemically precipitated using ammonia solution. A recovery rate of >90% was achieved for all three metals. Obtained metal oxides had a purity of 99% or more after the calcination process [28]. In all previous research, the pyrometallurgical process removed selenium in the first stage.

Although the above processes are developed at the laboratory level and need further development and adaptation to be employed as an industrial recycling process, these new approaches give an objective perspective to the economic recycling processes of the new generation of solar PV modules.

2.3 The recycling process and integration with the production of alloys for use in renewables

Figure 1 shows the material flowsheet for recycling from renewables and e-waste, with additional primary processing of the obtained metals in the same production plant, using the same equipment as for recycling processes and with the aim to utilize them in the renewable energy sector again.

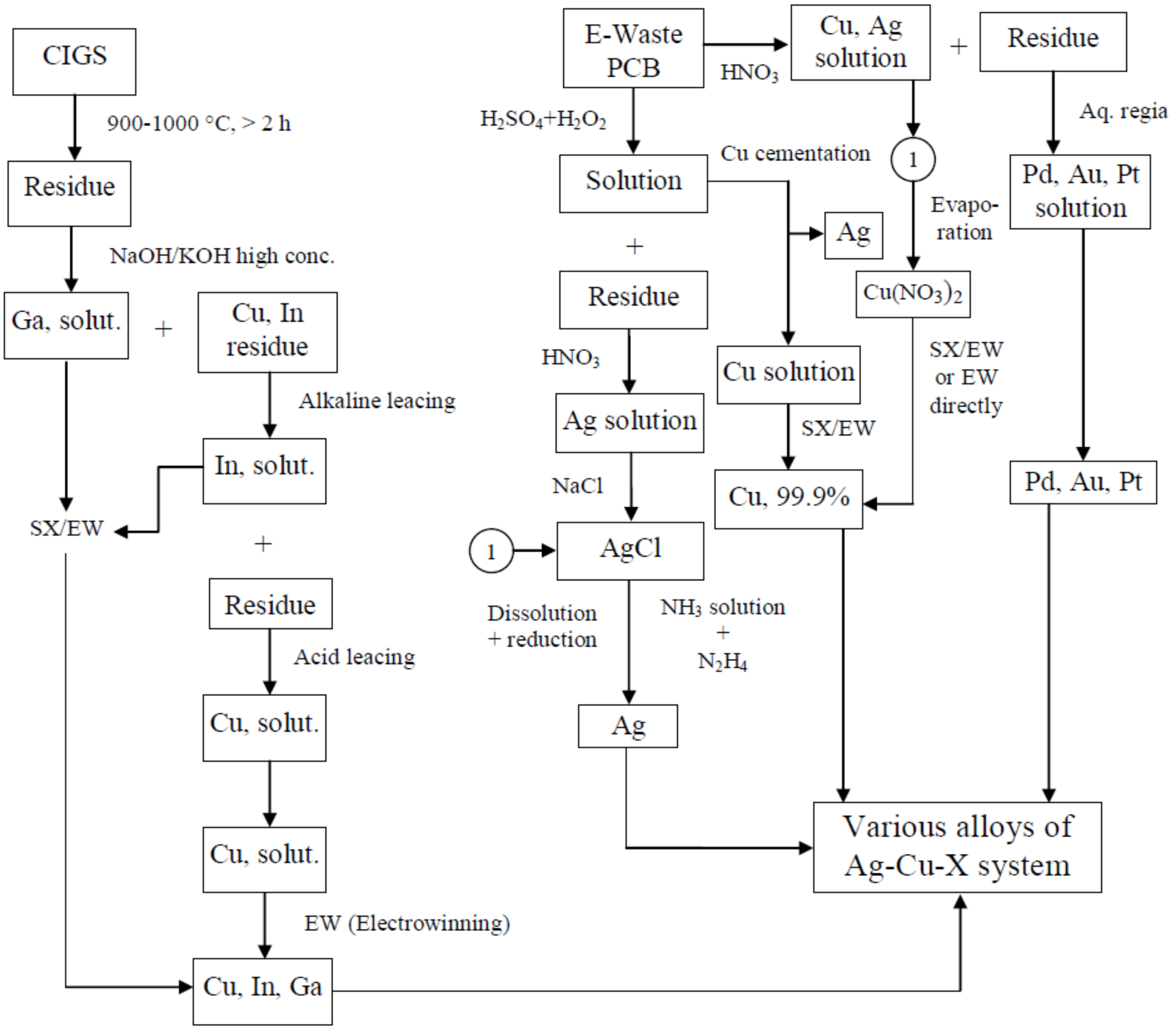

Figure 1. The integrated process of recycling CIGS and e-waste with alloy production

Most of the processes presented in figure 1 result from the team's research investigations and were tested on the laboratory or semi-industrial scale [29-31]. Recoveries and purity of the obtained metals from the operations in figure 1 were reasonably high and are given in table 1.

The pyrometallurgical phase preceded both hydrometallurgy routes for e-waste given in Table 1. It is significant in a way that slag from the process accounts for most of the losses of In and Ga. However, even with that, recoveries for these two metals are reasonably high. Other components have very high recycling rates for e-waste processing. Recycling of CIGS leads to high recoveries but requires complicated pretreatment to achieve such levels. Ga has a lower recovery rate than In only due to a lower content in the material and consequently lower concentration in the pregnant leach solution. The purity of copper can reach as high as 99.97% but also be closer to just 99% since not ideal conditions for the electrowinning process.

__Table 1.__ Obtained recovery for each of the recycled metal and their purity [11, 29-32]

Metal	Nitric acid/aqua regia (two phase) route		Sulfuric acid/nitric acid route		From Solar CIGS PV	
	Recovery rate, %	Purity, %	Recovery rate, %	Purity, %	Recovery rate, %	Purity, %
In	N/A	N/A	80.9	~99	Up to 96	99.9%
Ga	N/A	N/A	79.1	~99	~92	99.9%
Cu	95.6-97.1	99,97	92.4	99.7	89-93	≥99%
Ag	96.5-98,0	99,95	>95	98.8	N/A	N/A
Zn	92-95	98 (as ZnO)	N/A	N/A	N/A	N/A
Ni	94.6-95.7	>99 as sulfate	N/A	N/A	N/A	N/A

2.4 Alloys obtained from renewable and related electronics and their structure

To the extent of the paper, the most significant alloys obtained by the recycling process are those with applications in renewables. Regarding special attention to recovering critical elements - silver and indium, together with copper, alloys of the Ag-Cu-X system were the first choice for the application. Although not a critical metal, copper is a significant component because it has a high content in the waste, about 1% in CIGS PV panels and about 20-25% in PCB waste; additionally, recycled zinc was a component of the alloys, with 2-8% in e-waste and PCBs, respectively [31, 32].

Alloys of the Ag-Cu-X system with a high concentration of Ag (≥40%) and lower content of the third metal (under 30%), especially alloys with nearly 60 mass % of Ag, as presented in the paper ($Ag_{60}Cu_{26}Zn_{14}$, $Ag_{59}Cu_{31}Pd_{10}$, $Ag_{63}Cu_{27}In_{10}$, and $Ag_{40}Cu_{30}Zn_{30}$) are two-phase ternary alloys. They consist of silver rich phase (over 80% of silver) and Cu rich phase (varying from 65 to 95%); different third components tend to concentrate either in Cu (Pd, Zn) or Ag (In), depending on their solubility. The SEM images of alloys $Ag_{63}Cu_{27}In_{10}$ and $Ag_{40}Cu_{30}Zn_{30}$ are given in figures 2 and 3.

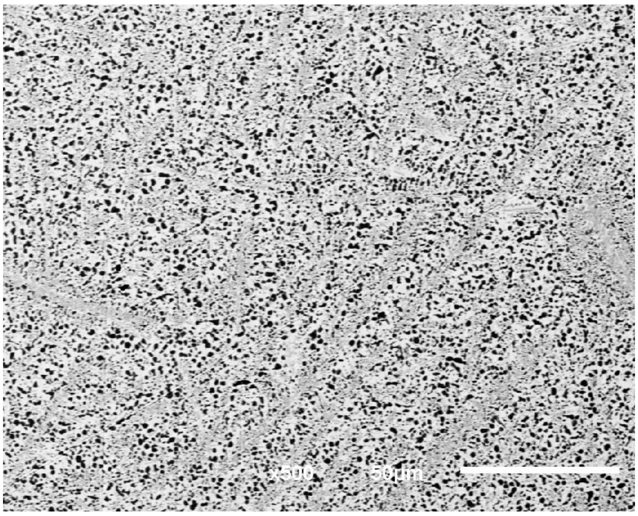

Figure 2. SEM micrgraph of $Ag_{63}Cu_{27}In_{10}$ alloy

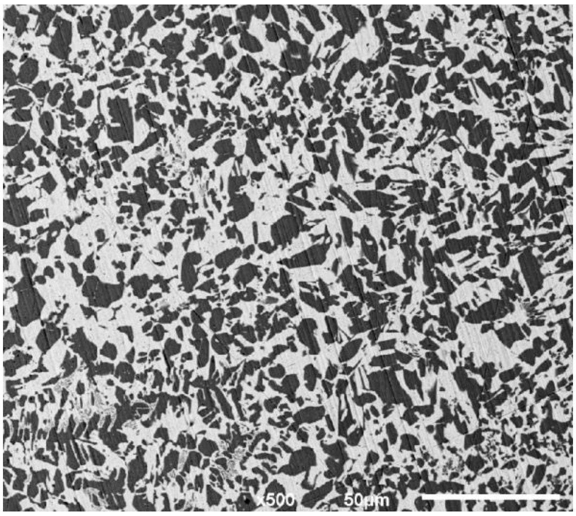

Figure 3. SEM micrgraph of $Ag_{40}Cu_{30}Zn_{30}$ alloy

Bright surfaces in figures 2 and 3 are Ag phase, and dark belong to Cu phase of the alloy. Microstructure details of the alloys can be different due to solidification conditions (here are given somewhat larger features than in the above papers, especially for $Ag_{40}Cu_{30}Zn_{30}$), but the two-phase structure is indubitable. Generally, phase composition can vary due to cooling rate (temperature dependence of the equilibrium in solid solution) but within relatively small differences, except for the quenching (rapid cooling). The detailed structure of the Ag-Cu-Zn alloys is given in paper with that theme [32], and the basic structure and detailed electrochemical characteristics of the palladium-bearing alloy together with $Ag_{60}Cu_{26}Zn_{14}$ have been also studied [33].

These alloys are widely used in renewable energy sources, especially in control electronics. They are used for joining very versatile materials, particularly very different kinds. Examples are joining copper and copper alloys with stainless steel, brazing cermets, and other metal-ceramics or ceramics materials to different metals, and filler for some rare metals [34]. Paper has shown advantageous integration of the alloys and recycling from renewables.

3 Conclusion

The importance of the recycling of renewable energy sources and the significance of the reuse of the recycling materials are highlighted in this paper.

The high cost of the recycling of some solar PV collectors and the deficiency of some critical elements offer a wide area for contemporary and future studies. It is shown that recycling rare metals provides an additional opportunity for cost-effectiveness by obtaining some components which have high value (Au, Pt), high content (Al), or both (Cu, Ni, Co) and are less insufficient.

The flowsheet based on earlier experimental work has given the principle for integrating e-waste and renewables. It is proposed to incorporate the principles of recycling and reuse within the same processes. The presented scheme combines all types of metallurgy, namely pyro-, hydro- and electro-, and uses various physical-chemistry methods like solvent extraction, oxidation-reduction reactions, and dissolving-precipitation.

The high recovery rates were obtained in those processes for base technical metals, mostly above 90%, and about 80% for technological metals (rare earth elements). The higher rates were for Cu (92.4-97.1%) and Ag (95-98%) and with exceptional purity, which was>99% for both of them. Finally, the structure of alloys of the Ag-Cu-X system has been presented to highlight their characteristics that qualify these materials for reuse for renewables and make the recycling process more economical and effective.

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