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Rapid removal of copper from wastewater by Fe-based amorphous alloy

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ABSTRACT

Fe-based amorphous alloys have been proved to exhibit excellent degradation properties for the removal of copper ions from simulated wastewater. Compared with crystalline iron, amorphous alloy has higher removal efficiency, lower reaction activation energy and high corrosion current density owing to its unique surface mobility. The copper ion can be reduced to less than 0.5 or 1 ppm at different initial concentrations. Even at a lower concentration of copper ions, it also exhibits better performance. In comparison, the concentration of the copper ion would be higher than the prescribed discharge standard if the wastewater was treated by crystalline iron. This work provides a potential application of Fe - based amorphous in the removal of heavy metal ions from wastewater.

1. Introduction

Our world is now facing many unprecedented environmental threats, one of which is water pollution [1–3]. Metals such as lead, cadmium, copper, arsenic, nickel, chromium, zinc and mercury are recognized as the hazardous heavy metals, resulting in damage to blood composition, lungs, kidneys, liver and other vital organs [2,4]. Even at low concentrations, the wastewaters containing these heavy metals still exhibit strong toxicity to human body [5,6]. In the past centuries, the world-wide rapid industrialization has greatly aggravated the release of these toxic heavy metals to water streams. At present, the discharge of contaminative wastewater is a common concern environmental problem of humankind. As the main sources of heavy metal ion contamination, electroplating, metal processing, printed circuit board (PCB) manufacturing, mining, textile and battery manufacturing industry are making this problem more and more serious [7,8].

Among the heavy metals, copper ion (Cu^{2+}) is a common hazardous pollutant in wastewater and is often released by metallurgical, plating, printing circuits, fertilizer and refining industries [9,10]. Though Cu^{2+} is known to be a necessary trace element [11–13], but it can accumulate in living organisms, causing several disorders and diseases [14–17]. According to the regulations of world health organization (WHO), the guideline value for Cu^{2+} in drinking-water is 2.0 mg/L [13]. However, the concentration of the copper ion in the industrial effluent ranges from tens to even thousands mg/L in different industrial fields, which is tremendously far higher than the regulations. Furthermore, when the concentration of Cu ion becomes very low, it will be very costly to decrease the concentration by traditional methods including electrochemical operations, reverse osmosis, or chemical treatment processes [18–22].

Metallic glasses, alternatively known as amorphous alloys, including Fe- [23-26], Mg- [27], Al- [28] and Co- [29] based amorphous alloys have been very interesting catalytic materials for degrading the water contaminants due to the intrinsic difference of their disordered atomic packing arrangement compared with the well-defined atomic ordering in crystalline materials [23,24,30–33]. In recent experiments, it was discovered that the surface mobility is very different from the bulk in amorphous matters owing to the disordered structure [34], including glassy polymers [35], oxide glasses [36], organic glasses [37], amorphous alloys [38,39] et al. Evidences showed that the surface diffusion was dominated by the mono-atomic layer for the crystalline solid, however, as for the amorphous matters, the domination layer could be as thick as nanometer [40,41]. Specifically, the surface diffusion rate of amorphous alloy is millions of times higher than the bulk when subject at temperature below glass transition temperature. That is to say, there exists a "super active" layer with high mobility on the surface of amorphous alloys [38]. Inspired by such fast surface mobility of amorphous alloy, in this work we present the rapid removal of copper ions from wastewater could be achieved by the Fe-based amorphous alloy ribbons. Furthermore, by using such method, no secondary pollution is caused, the resultant of reaction is pure copper, which can be collected and reused. Therefore, it is proved to be an efficient and cost-saving method to prevent the copper ion pollution in wastewater, showing great significance in the sense of environmental protection.

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2. Materials and methods

2.1. Materials and characterization

The Fe₇₈Si₉B₁₃ (at.%) amorphous alloy ribbons with a thickness of 25 μ m were chosen for this work. The amorphous nature of the Fe-based amorphous alloys were ascertained by x-ray diffraction (XRD; Rigaku MiniFlex600) with Cu K\alpha radiation and differential scanning calorimetry (DSC; Perkin–Elmer DSC-8000) at a heating rate of 20 K/min. The micro morphologies of the moulds and the punched products were collected on a scanning electron microscope (SEM; FEI QUANTA FEG 450) instrument. The electrochemical tests were conducted using a CHI 660E electrochemical analyzer (CH Instruments, Inc., Shanghai) in a standard three-electrode system, using a platinum plate as the counter electrode, and a saturated calomel electrode (SCE) as the reference electrode. The electrochemical measurement area was defined as 1 cm².

2.2. Wastewater containing Cu ion

The simulated wastewater containing Cu ion was manually designed by dissolving corresponding quantities of $CuSO_4$ in distilled water, preparing into different concentrations. In present research, the initial concentrations of Cu^{2+} are 500 ppm (i. e. mg/L), 200 ppm, 100 ppm, 50 ppm and 10 ppm. The concentration of copper ions in the solution was determined by Inductively Coupled Plasma-Optical Emission Spectrometry(ICP-OES; PerkinElmer Optima2100DV).

2.3. Experiment of the Cu ion removal

An amount of 300 ml copper ion solution (100 ppm if not noted) was prepared in a beaker. The solution was stirred and mixed by a mechanical agitator throughout the copper removal process. The pH value of the treated solution was adjusted to 2 by 10% H_2SO_4 and 10% NaOH. The temperature (25 °C if not noted) of the aqueous solution containing copper ions was maintained using a thermostatic water bath. After that, 0.6 g of amorphous alloy ribbons (2 g L⁻¹ if not noted) were added into the solution and the reaction time was recorded. At regular intervals, 5 ml of the solution was removed using a pipette and filtered with a 0.45 μ m filter membrane. The concentration of copper ions in the filtrate was determined by the ICP-OES.

3. Results and discussion

3.1. Copper iron removal by amorphous alloy

It should be noted that the $\mathrm{Fe}_{78}\mathrm{Si}_9\mathrm{B}_{13}$ ribbons were amorphous before the wastewater treatment, the XRD pattern and DSC curve were the evidences, which are presented in Figs. S1(a) and (c). The crystalline iron ribbons had a typical crystal x-ray diffraction pattern Fig. S1(b). The basic principle of copper ion removal can be schematically illustrated in Fig. 1(a). As it is shown, the amorphous alloy ribbon was immersed into the wastewater, after a period of time, the wastewater got clear and the crystal copper layer applied on the surface of the ribbon. Fig. 1(b) shows the color contrast before and after cooper ion removal of the wastewater. Obviously, the color of the wastewater changed from light green to colorless and transparent, indicating the copper ion removal effect. Fig. 1(c) presents the comparison of the original ribbon and the one after copper ion removal. The clear pure copper layer can be seen, and the inset in Fig. 1(c) shows the pure copper that was scraped from the amorphous alloy ribbons or collected through a simple filtration of the treated wastewater solution, therefore, the copper could be collected and reused, no follow-up treatment is needed. In addition, the copper is pure and without other impurities that can be demonstrated from the copper XRD pattern Fig. S1(d). The video in the supplementary materials (fast forward mode) compares the color change of the amorphous ribbon (right) and iron ribbons (left) in the solution containing Cu ion of 500 ppm. Clearly, the copper layer coated on the amorphous ribbon in a short time (less than 2 min), in contrast, nearly no change can be



Fig. 1. (a) Schematic diagram of the copper removal process. (b) The contrast of the wastewater before and after copper ion removal treatment. (c) The comparison of the original ribbon and the one after copper ion removal. The inset shows the pure copper that was scraped from the amorphous alloy ribbon.

observed for the iron. The results demonstrate that the amorphous ribbon has excellent Cu ion displacement ability compared with the crystalline alloy. This ability makes it ideal candidate for the industrial application in the fast and efficient removal of the Cu ion in wastewater.

3.2. Uptake efficiency of the removal process

To precisely analyze the concentration change in the copper removal reaction, we define the uptake efficiency of the copper ion as η , which can be calculated using the following equation:

$$\eta = \left[(C_o - C_t) / C_o \right] \times 100 \tag{1}$$

where C_0 and C_t are the initial and the instant concentration at time t of copper ion in the wastewater. Fig. 2 shows the reactivity of amorphous alloy ribbons and crystalline iron ribbons in treating copper ionic solutions with different concentrations. Comparison in Fig. 2(a) and (b) indicates that the amorphous alloy has better reaction performance than the crystalline iron at different concentrations of copper ions. The amorphous alloy can remove the copper ion concentration to less than 1 ppm at different initial concentrations of copper ions in the experimental time as shown in Fig. S2. These results are satisfactory with the Chinese integrated wastewater discharge standard for the Cu ion content of wasterwater (GB8978-1996).But it is a pity that crystalline iron lacks such excellent performance. Fig. 2(a) shows that amorphous alloys need more time to remove high concentrations of copper ions than low concentrations. When the concentration of copper ions in the solution increased from 10 ppm to 50 ppm, 100 ppm, 200 ppm and 500 ppm, the time to achieve the experimental purpose increased from 80 min to 80 min, 100 min, 160 min and 180 min, respectively. Whereas for crystalline iron in Fig. 2(b), the final concentration of copper ions in the solution was greater than 3 ppm (as shown in Fig. S3), even in the group of 10 ppm for 180 min. As for the iron, it should be noted that the concentration of copper ions in the solution decreased at the beginning of the experiment and then increased. Fig. 2(c) and (d) show how the corresponding uptake efficiency η of the amorphous alloy and crystalline iron change with time. The η of amorphous alloy is 99.8% higher than crystalline iron 96.3% at 500 ppm of copper ionic solution, but amorphous alloy is 97.0%, crystalline iron 65.8% at 10 ppm. And the η increases as the copper ion concentration increases.

3.3. Reaction activation energy

The unique copper ion removal ability in the wastewater can be ascribed to its more active sites originates from the intrinsic chemical heterogeneity on the disordered surface than the crystalline alloy. As it is stated above, the surface mobility of amorphous materials is very different from the crystalline. The surface diffusion coefficient is millions faster than the bulk which has been found in previous work [38, 40-42]. For amorphous materials, the active atomic layer could be as thick as nanometer range, i. e. several atomic layers, however, such surface dynamics is limited to mono atomic layers for crystalline solids. Benefiting from this property, the reaction activation energy of amorphous ribbon could be much lower than the crystalline alloy in dealing with the wastewater. To confirm that, the thermal activation energy of copper ion removal using corresponding ribbons is analyzed by carrying out the reactions at different temperatures, as illustrated in Fig. 3. Fig. 3 (a) and (b) show the effect of temperature on the removal of copper ions from the solution. Clearly, the removal process takes less time with an increased solution temperature. The increase of temperature intensifies the molecular thermal motion and promotes the electron transport of the reactive material, therefore, resulting in the higher removal efficiency. The concentration of copper ions in the solution was decreased below 1 ppm with an amorphous alloy within 60-100 min at 298.15-328.15 K. However, it is clear that crystalline iron is difficult to effectively remove copper ions compared with the amorphous alloy. According to the ln



Fig. 2. The copper ion concentration changes with time in the removal process using (a) $Fe_{78}Si_9B_{13}$ and (b) crystalline iron at different concentrations of copper ions; the uptake efficiency η of the amorphous alloy (c) and crystalline iron (d) change with time.



Fig. 3. The concentration change of copper ion solution in the removal process using (a) $Fe_{78}Si_9B_{13}$ and (b) crystalline iron at different temperature; the $ln(C_0/C_t)$ vs. time curves for (c) $Fe_{78}Si_9B_{13}$ and (d) crystalline iron. (e) The reaction rate constants k at different reaction temperatures; (f) ln k vs. -1/RT curves for $Fe_{78}Si_9B_{13}$ amorphous alloys ribbons and crystalline iron ribbons.

 (C_0/C_t) vs .time curve, the process of removal reaction follows a first-order reaction kinetics equation, with the goodness of all linear fit R^2 are greater than 0.998 as shown in Fig. 3(c) and (d). Fig. 3(e) summarizes the reaction rate constant *k* at different temperatures of amorphous ribbon and the iron ribbon, respectively. The reaction rate constant of amorphous alloy increases from 0.04886min⁻¹ to 0.0667min⁻¹,0.08349 min⁻¹ and 0.09833 min⁻¹, when the solution temperature increases from 298.15 K to 308.15 K, 318.15 K and 328.15 K, while that of crystalline iron increases from 0.01338min⁻¹ to 0.01945min⁻¹,0.02710 min⁻¹ and 0.03438 min⁻¹. It is noticed that the reaction rate constant of amorphous alloys is 3 times higher than

crystalline materials, indicating the great potential of amorphous alloy in dealing with the wastewater containing heavy metals.

The reaction conditions are set as follows: initial pH = 2, ribbon dosage 2.0 g L⁻¹ and $C_0 = 100$ ppm. The reaction kinetics is commonly described by the pseudo-first-order equation as given below:

$$C_t = C_0 \exp(-kt) \tag{2}$$

where *k* is the reaction rate constant (min⁻¹), *t* is the reaction time (min), C_0 is the initial concentration of copper ion (ppm), and C_t is the instant concentration (ppm) at time *t*. Therefore, the reaction rate constant *k* can be derived as follows:

$$k = \ln(C_0 / C) / t \tag{3}$$

The thermal activation energy for the copper ion removal can be calculated by the Arrhenius-type equation:

$$\ln k_T = -\Delta E/RT + \ln A \tag{4}$$

where k_T is the reaction rate constant at different temperatures, ΔE is the reaction activation energy, *R* is the gas constant and *A* is a constant. Fig. 3(f) plotted ln k_T vs.-1/RT and it is calculated that the reaction activation energies of amorphous alloy and iron ribbons are 18.95 kJ mol⁻¹ and 25.78 kJ mol⁻¹, respectively. Apparently, the amorphous alloys with low reaction activation energy have better performance than the iron ribbons.

3.4. Electrochemical property

In the reported study, the Fe - based amorphous alloy has excellent stability in the degradation of organic dyes. The stable service life of amorphous ribbons for degradation application is related to their corrosion resistance [23,43]. Unlike catalytic reactions, the displacement reaction of copper ion is a spontaneous redox reaction. Thus, the corrosion properties of iron and Fe78Si9B13 amorphous ribbons in 100 ppm copper ion solution were compared. Open circuit potential and polarization curves of the above ribbons are shown in Fig. 4. After 30 min, the open circuit potential of Fe78Si9B13 amorphous ribbons is 0.639 V and iron is 0.617 V as shown in Fig. 4(a). Iron and Fe₇₈Si₉B₁₃ amorphous ribbons have similar open circuit potentials. It is possible that elemental iron is corroded on amorphous and iron surfaces. But, the corrosion current density of $Fe_{78}Si_9B_{13}$ amorphous ribbons are 1.43 \times 10^{-4} Acm⁻², which is higher than 4.71×10^{-5} Acm⁻² for iron as shown in Fig. 4(b). The high corrosion current density indicates that more iron atoms are dissolved and copper ions are reduced. It also means that Fe based amorphous alloys have the ability to quickly remove copper ions. This is most likely due to the unique disordered atomic structure of amorphous alloys.

3.5. Mechanism of the copper removal process

As it was stated in the introduction, researchers have found that the surface mobility is very different from the bulk in amorphous matters, the surface diffusion rate of amorphous alloy is much higher than that of crystalline solid, which means an active "liquid like" layer exists on the surface of amorphous alloy [39]. Dynamic modulus mapping (DMM) of $Fe_{78}Si_9B_{13}$ amorphous alloy ribbons and crystalline iron ribbons as shown in Fig. 5(a). Compared with iron ribbons, the surface of $Fe_{78}Si_9B_{13}$ amorphous ribbons have active kinetic behavior due to low modulus distribution. At the same time, the high corrosion current density of amorphous alloy materials can also explain the active corrosion dynamic characteristics. Fig. 5(b) compare the mechanism of the

copper ion removal process of amorphous alloy ribbons and iron ribbons. The Fe-based amorphous alloy ribbons were prepared by rapidly cooling the molten liquid alloy on a high-speed rotating copper mold. In this process, the disordered atomic structure of the alloy was retained. In particular, there are active atomic layers on the free surface of amorphous alloys, which are similar to liquid atoms with excellent reactivity. The electrons from iron atoms in the active atomic layer can be rapidly transferred to copper ions when the amorphous alloy ribbons were placed into a copper ion solution. In addition, abundant galvanic cells were produced due to the iron and copper, which was beneficial to the reaction process. However, the surface mobility of the crystalline iron ribbons is much weaker than the amorphous alloy due to the stable thermodynamic state. Consequently, the efficiency of removing copper ions with crystalline iron ribbons are lower than that amorphous alloy ribbons.

4. Conclusions

In summary, we demonstrated that the amorphous alloys ribbons exhibit highly efficient removal of copper ions from wastewater. It could reduce the concentration of copper ions in the solution to less than 1 ppm, not only the metal copper is obtained from the solution, but also the treatment of wastewater can reach the discharge standard. This property is suitable for high or low concentration copper ion wastewater. Compared with the activation energy of amorphous alloy ribbons and iron ribbons, the amorphous alloy could provide more active sites for the reaction owing to its unique surface atomic mobility, so it has low reaction activation energy and high corrosion current density. This work not only provides an efficient and low-cost solution for remove the copper ions from the solution, but also expands the application areas of Fe-based amorphous alloys.

Declaration of competing interest

We would like to submit the enclosed manuscript entitled "**Rapid removal of copper from wastewater by Fe-based amorphous alloy**", which we wish to be considered for publication in *Intermetallics*. No conflict of interest exits in the submission of this manuscript, and manuscript is approved by all authors for publication. I would like to declare on behalf of my co-authors that the work described was original research that has not been published previously, and not under consideration for publication elsewhere, in whole or in part. All the authors listed have approved the manuscript that is enclosed.

CRediT authorship contribution statement

Yuqiang Yan: Conceptualization, Data curation, Formal analysis. Xiong Liang: Investigation, Methodology, Project administration. Jiang Ma: Supervision, Resources, Writing - original draft, Writing - review &



Fig. 4. The open circuit potential(a) and polarization curves(b) of iron and Fe₇₈Si₉B₁₃ amorphous ribbons in 100 ppm copper ion solution.



Fig. 5. (a) Dynamic modulus mapping (DMM) of $Fe_{78}Si_9B_{13}$ amorphous alloy ribbons and crystalline iron ribbons. The scale bars are identical and equal 100 nm. (b) Schematic diagram of removal copper ions by amorphous alloy ribbons and crystalline iron ribbons.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.intermet.2020.106849.

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