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In Situ Wire + Powder Synchronous Arc Additive Manufacturing of Ti–Cu Alloys

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Abstract

In this study, a new wire + powder synchronous arc additive manufacturing technique was used to manufacture Ti–Cu alloys. The microstructure and properties of the as-fabricated alloys were studied. The results showed that the prepared Ti–Cu alloys have good properties. The Cu with high growth restriction factor can increase the constitutional supercooling zone in the Ti–Cu alloys, which can override the negative effect of a high thermal gradient in the manufacturing process. Through the observation of the microstructure, the as-printed Ti–Cu alloy specimens have equiaxed fine-grained microstructure. Through corrosion performance analysis, the Cu can also make the passivation film of the alloy more compact and make the alloy more corrosion resistant.

Keywords: additive manufacturing, wire+ powder, Ti-Cu alloys, mechanical properties, corrosion

Introduction

ADDITIVE MANUFACTURING (AM) PROVIDES an effective method to fabricate complex structural components with multiple materials.^{1–4} Wire–arc additive manufacturing (WAAM) is a cheaper AM method than electron beam melting and selective laser melting methods.^{5–7} WAAM offers a high arc column temperature, high deposition efficiency, and high handling mobility, which is suitable for manufacturing large structures with multiple materials.^{8–10} However, alloys produced by WAAM have low surface precision.^{11,12} The surface precision of the samples produced by powder feeding arc AM is high with the appropriate process and parameters, but the utilization rate of powder is low, which easily causes dust pollution.^{13–15}

In view of the mentioned analysis, we propose wire + powder synchronous arc AM. Combined wire + powder synchronous arc AM is based on an arc as the heat source, and wire and powder material are simultaneously filled in the molten pool. The deposition is performed layer by layer to achieve AM. It is a new AM technology. Wire + powder

synchronous arc AM combines the advantages of the two technologies.

It has the advantages of high efficiency, high density, and flexible adjustment and is suitable for the additive preparation of metal matrix composites and functionally graded materials.^{16–18} The addition of wire is conducive to increasing the area of the deposited layer, providing a way to manufacture complex structural parts and improving the preparation efficiency and material density. The addition of powder can provide a reinforcing phase, and a metal composite material with a gradient composition of the particle reinforcing phase can be prepared.

More importantly, the wire + powder synchronous arc AM can inhibit the segregation that Ti–Cu alloys easily exist during the solidification process, so it is favorable for making Ti–Cu alloys. A new method for preparing titanium alloy has been developed.¹⁹

Ti–Cu alloys have excellent corrosion resistance, strong antibacterial properties, and high strength, which are widely used in the petroleum, chemical, metallurgy, biomedicine, and aerospace industries.²⁰ For Ti–Cu alloys, the addition of

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FIG. 1. Illustration of the wire + powder synchronous arc additive manufacturing setup (a), illustration of deposition (b), and as-deposited Ti-7.3%Cu sample (c).

Cu provides a wider β -phase transition zone on the Ti-rich side of the Ti–Cu phase diagram, which can form various forms of intermetallic compounds so that they have different features to meet the application of different occasions.^{20–25} Although Ti–Cu alloys are a very promising material, the casting of Ti–Cu is particularly difficult due to its high melting temperature and high reactivity with oxygen, and it is prone to segregation and dendrites during casting.

Moniri Javadhesari et al. and Liu et al.^{26,27} studied the relationship between copper content and antibacterial property of Ti–Cu alloys. The content of copper had a great impact on antibacterial properties, but did not study the effect on mechanical properties. Hayama et al., Holden et al., and Williams et al.^{20,28,29} studied the effect of heat treatment on the mechanical behavior of Ti–Cu alloys. Their research showed that the cooling rate affects the phase transformation, which had a major impact on mechanical properties. These studies give us two inspirations. If we want to obtain high performance Ti–Cu alloys, we can start with copper content and technology.

Metal AM process has two main characteristics: high cooling rates and high thermal gradient, which often lead to two completely different phenomena: one is very fine microstructure, and the other is almost exclusively columnar grains.³⁰ Cu as a solute element has high growth restriction factor Q, and a supercooled zone can be formed. According to interdependence theory, if the composition of the supercooled zone is larger, the grain will be easier to refine.³¹ Therefore, using this method to manufacture Ti–Cu alloys is more prone to fine equiaxed grains. It solves the segregation in the casting process and the inherent large columnar crystal defect in AM.

In this research, wire + powder synchronous arc AM was used to manufacture Ti–Cu alloys. We used pure Ti wire (\geq 99.9%) and pure Cu powder (\geq 99.99%) as raw materials. Ti wire is used to provide a dense titanium matrix, and Cu powder is used to refine the grain and increase the reinforcing phase. We studied the properties of Ti–7.3%Cu alloys prepared by wire + powder synergistically by controlling the wire feeding speed and feeding amount.

Experiment

Process and equipment introduction

The wire + powder synchronous arc AM setup is shown in Figure 1a. The AM process is shown in Figure 1b. It mainly includes powder plasma arc additive manufacturing (PPA-

 TABLE 1. WIRE + POWDER SYNCHRONOUS ARC ADDITIVE

 MANUFACTURING PROCESS PARAMETERS

Parameter	Value	Unit
Deposition current	105	А
Pulse period	63	μs
Cu powder feed velocity	0.7	g/min
Ti wire speed	8.9	g/min
Travel speed	400	mm/min
Shielding Ar gas	10	L/min
Powder feeding Ar gas	3.5	L/min
Dwell time between deposition layers	2	min
Angle between the torch and filler wire	60	0
Distance between the torch and workpiece	15	mm



FIG. 2. Schematic showing the location in the Ti-Cu alloys from which the test samples were extracted.



FIG. 3. Optical micrographs of the Ti6–Al4–V substrate (a) and Ti–7.3%Cu alloys (b).

AM) and a wire feeder (WPC-600 feeder machine). The purity of Cu powder (100 mesh size) was >99.9%, which was fed by a powder feeder machine. The purity of Ti wire (\geq 99.9%) with a diameter of 1.2 mm, which was fed by a WPC-600 feeder machine. Ti–Cu alloys composition control is achieved by adjusting the Cu powder feeding amount and Ti wire feeding speed. After many experiments, we selected the best process parameters, as given in Table 1.

The substrate was a $150 \times 100 \times 10$ mm rolled Ti6–Al–4V plate. The as-fabricated sample is shown in Figure 1c. The samples were deposited in a total of four layers with a total height of 15 mm. Owing to the heating instability caused by arc retracting, the fuse is not timely, which makes the two ends of the sample forming quality is not good.

The locations of the printed Ti–Cu alloys from which samples were taken for analysis are shown in Figure 2. The microhardness was measured by an HV-1000 microhardness tester. The phase composition in Ti–Cu alloys was analyzed using X-ray diffraction (XRD). The metallographic sample was etched with hydrofluoric acid solution, and the microstructure was observed by a scanning electron microscope. The grain behavior was studied by electron backscattered diffraction (EBSD). One electrochemical impedance spectroscopy (EIS) electrochemical workstation was used to study the corrosion behavior at room temperature. The 3.5% NaCl corrosion solution was used. The corroded surface was analyzed using a 3D laser microscope OLS4100.

Results and Discussion

Microstructure

The microstructures of the Ti–7.3%Cu alloys and Ti–6Al– 4V substrate are shown in Figure 3. Figure 3a shows that there is slightly elongated α grains (light) and intergranular β grains (gray) in the Ti6–Al4–V substrate. The Ti6–Al4–V substrate is cast and rolled. It can be seen from Figure 3a that the structure of the substrate is fine and evenly distributed.



FIG. 4. X-ray diffraction patterns of Ti-7.3%Cu alloys.



FIG. 5. EBSD inverse pole figures (a), and the grain size distribution (b). EBSD, electron backscattered diffraction.

Figure 3b shows the fine and equiaxed α + Ti₂Cu grains. When the temperature is 792°C, β -Ti will transform into α -Ti and Ti₂Cu phases. It is consistent with the XRD test results (Fig. 4).

In general, large columnar crystals will inevitably appear in AM.³² However, the Ti–7.3%Cu alloys produced by wire + powder synchronous manufacturing have not only large columnar crystals but also fine grains and some equiaxed crystals. To further analyze the microstructure, the EBSD test was performed on the sample, and the results are shown in the Figure 5. The microstructure presents a basket network, and there is also a lamellar structure in the microstructure, and it can be clearly seen that there are fine equiaxed structures (Fig. 5a). According to the statistics of grain size, most grain sizes are <10 μ m (Fig. 5b).

Stjohn et al.³¹ found the constitutional supercooling zone is the main factor affecting the grain size according to interdependence theory. The formation of microstructure can be analyzed in combination with Equation (1):

$$\frac{G}{R} \ge \frac{m_l C_0(K-1)}{k D_l},\tag{1}$$



Hardness

The bottom is the starting point, and the top is the end point to test the hardness of the alloy. The following hardness results are plotted based on the average results and the error values (Fig. 6). The hardness is measured every 1.5 mm. Six points were detected at the substrate, and eight points were detected at the deposition layer. The average hardness







FIG. 7. Representative engineering stress–strain curves of the Ti–7.3%Cu alloys.

TABLE 2. MECHANICAL PROPERTIES OF TI-CU ALLOYS

Alloys (wt.%)	YS (MPa)	UTS (MPa)	e (%)
Ti–7.3Cu	$655 \pm 20 \\ 522 \pm 46$	800 ± 10	2.1 ± 0.2
Ti–7.1Cu (Ref. ²⁰)		660 ± 52	3.5 ± 0.8

 ε , uniform elongation; UTS, ultimate tensile strength; YS, yield strength.



FIG. 8. Potential dynamic curve.

TABLE 3. ELECTROCHEMICAL DATA OF THE TI-7.3%CUSAMPLE OBTAINED FROM THE POLARIZATION CURVE

Sample	E_{corr} (mV)	E_{pit} (mV)	I_{corr} ($\mu A/cm^2$)
Ti-7.3%Cu	-415	275	0.21

 $E_{\rm corr},$ corrosion potential; $E_{\rm pit},$ breakdown potential; $I_{\rm corr},$ corrosion current density.

obtained for the substrate was 346 ± 15 Hv. For the Ti-7.3%Cu alloy, the average hardness value increased by 6.6% (369 ± 10 Hv). The hardness values are between those of the cast Ti-7.1%Cu (330 ± 20 Hv) alloys and the Ti-15%Cu (380 ± 30 Hv) alloys.²⁰

Tensile properties

The corresponding tensile results are shown in Figure 7. The 0.2% offset yield strength, ultimate tensile strength, and uniform elongation (ε) are given in Table 2. It can be seen from the results that the sample has high strength, but low ductility. Its performance is better than that of Ti–7.1Cu alloy cast and heat treated at 900°C for 2 h. Comparing the Ti–7.1 Cu, it has higher strength, that is mainly due to the higher volume fraction of eutectoid lamellae α -Ti, as shown in Figure 4. It has the same ductility as cast Ti–7.1 Cu alloys. This is mainly due to the very fine structure of the alloys produced by this method and the achievable transformation of columnar crystals into equiaxed crystals.

Corrosion resistance

The mentioned analysis shows that the Ti–7.3%Cu alloys made by wire + powder synchronous arc AM had good mechanical properties. Now, the corrosion property of the Ti– 7.3%Cu alloys is studied. Figure 8 shows the potentiodynamic polarization curve. The Ti–Cu alloy has an obvious passivation phenomenon in NaCl solution, and the passivation zone is wide, so it has good corrosion resistance. The electrochemical parameters, including corrosion potential (E_{corr}), breakdown potential (E_{pit}), and corrosion current density (I_{corr}), are given in Table 3.

With respect to E_{corr} , the Ti–7.3%Cu sample showed a more positive E_{corr} (430 mV) than commercial pure-Ti.³³ Cu can make the passivation film more compact and make the alloy more corrosion resistant. The alloy prepared by wire + powder synchronous arc AM not only has good mechanical properties but also good corrosion resistance.

EIS measurements are performed, and the results are shown in Figure 9. Figure 9a shows the Nyquist diagrams, and Figure 9b and c shows the Bode diagrams. Figure 9b and c shows that the absolute impedance curve in the high-frequency regime (10^4-10^5 Hz) is nearly always related to the phase angle close to 0. This is a typical solution resistance (R_s) characteristic. From 10^3 to 10^{-1} Hz (medium and low frequency), there are two time constants, as shown in Figure 9b. This may be a two-layer structure of passive film.

As titanium is relatively active, it is easy to form oxides, and according to the report by Roh and Macdonald,³⁴ when



FIG. 9. Nyquist plot diagram (a), Bode phase (b), and Bode plot diagram (c).



Layer Layer

FIG. 10. Equivalent circuit used for fitting the EIS data. EIS, electrochemical impedance spectroscopy.

TABLE 4. EQUIVALENT CIRCUIT PARAMETERS FOR THE ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY SPECTRA OF THE TI-7.3%CU SAMPLE

Parameters	Ti–7.3%Cu
$\overline{\text{Rs}(\Omega;\text{cm}^2)}$	12.82
CPE_1 (F/cm ²)	1.117×10^{-5}
$R_1 (k\Omega \cdot cm^2)$	4.618
n ₁ , hydroxide layer	0.98
CPE_2 (F/cm ²)	3.818×10^{-5}
$R_2 (k\Omega \cdot cm^2)$	37.17
n ₂ , hydroxide layer	0.61
Chi-squared, χ^2	6.68×10^{-3}

titanium alloy is corroded, two layers of passive film with barrier (inner) layer and outer hydroxide layer will be formed. Based on the mentioned characteristics, an equivalent circuit with two time constants (Fig. 10) was used with ZView[®] software to fit the data. R_s indicates the resistance of the solution in equivalent circuit. The charge transfer resistance of the hydroxide layer is R_1 and the charge transfer resistance of the barrier layer is R_2 . The impedance of the hydroxide layer is CPE₁.

The barrier layer is a component of the double layer passive film and its impedance is CPE₂. By comparing the actual results with the fitting results, the error is within 10%, which shows that the results are reliable. The simulated electrical circuit parameters are summarized in Table 4. From Table 4, the resistance and capacitance of the barrier layer are greater than the resistance and capacitance of the hydroxide layer. This is mainly because the barrier layer formed on the metal surface is relatively stable, whereas the oxide film is unstable, and the oxide film is easily damaged by the electrochemical reaction. The R₂ value reaches 37,170 $\Omega \cdot cm^2$, and the CPE₂ value is very small, indicating that the passive film formed is relatively dense and has good corrosion resistance.

To see the surface morphology after corrosion more intuitively, we used a 3D topography instrument to observe the corrosion surface. Figure 11a shows that the surface has been corroded, and the corrosion severity of the entire sample surface is similar, which also indicates that the sample composition is relatively uniform. The number and size of pits are relatively small (Fig. 11b). The seriously corroded parts often form pits, and the expansion of corrosion is the main reason for the change in pit size. The root mean square roughness (Rq) of the surface is $1.106 \,\mu\text{m}$. The small height fluctuations shown in Figure 11c also confirm this. The fluctuation amplitude of the selected area is $2.85 \,\mu\text{m}$.



FIG. 11. Surface morphology of the corrosion sample (a), corresponding 3D topography (b), and amplitude curve of the selected section surface height (c).

Conclusions

In this study, a new wire + powder synchronous arc AM method is proposed to make a Ti–Cu alloy. A sample with a smooth surface and good forming quality was successfully deposited. The microstructure, hardness, tensile properties, and corrosion resistance of the Ti–7.3%Cu alloys were investigated. The conclusions of this article are as follows:

- (1) This method shows great manufacturing potential. The Ti–Cu alloys prepared in this study have no cracks and pores and its properties are better than those of casting alloys.
- (2) Most of the Ti–Cu alloys prepared by this method have a grain size of $10 \,\mu$ m, and have equiaxed crystal formation, which overcomes the generation of large columnar crystal in AM and provides a method to solve the inherent large columnar crystal in AM.
- (3) In the research, an equivalent circuit with two time constants is used. The barrier layer formed on the metal surface is relatively stable, whereas the oxide film is unstable, and the oxide film is easily damaged by electrochemical reactions. The copper can make the passivation film more compact and make the alloy more corrosion resistant.

Authors' Contributions

All authors have seen and approved the final version of the article. The article is the author's original work and has not been previously published and is not under consideration for publication elsewhere. C.S. and Y.W. contributed equally to this study. C.S. and Y.W. contributed to conceptualization, investigation, methodology, and writing—original draft. W.W. and L.H. tested samples. S.K. was involved in supervision and writing—review and editing. X.C. was involved in supervision and funding acquisition. S.Q. carried out tests and analysis of EBSD, review, and editing.

Author Disclosure Statement

No conflict of interest exists in the submission of this article, and the article is approved by all authors for publication.

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