Microstructure and properties of TP2 copper tube with La microalloying by horizontal continuous casting

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Abstract: The TP2 copper tube was prepared with La microalloying by horizontal continuous casting (HCC). The absorptivity of La and its effects on microstructure, tensile and corrosion properties of HCC TP2 copper tube were studied by means of the inductively coupled plasma optical emission spectrometer (ICP-OES), optical microscope (OM), scanning electron microscope (SEM) and potentiodynamic polarization measurements. The results show that the absorptivity of La in the HCC TP2 copper tube is about 15% under antivacuum conditions due to the good chemical activities of La. The impurity elements in copper tube such as O, S, Pb and Si can be significantly reduced, and the average columnar dendrite spacing of the copper tube can also be reduced from 2.21 mm to 0.93 mm by adding La. The ultimate tensile strength and the elongation with and without La addition are almost unchanged. However, the annual corrosion rate of the HCC TP2 copper tube is reduced from 10.18 mm·a⁻¹ to 9.37 mm·a⁻¹ by the purification effect of trace La.

Key words: rare earth; microstructure; mechanical properties; corrosion resistance; HCC TP2 copper tube

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Owing to the superior electrical and thermal conductivity, good mechanical properties and processability, as well as favorable corrosion resistance, pure copper, especially the phosphorus deoxidized copper (TP2), has been widely used for air-conditioning and refrigeration (ACR) tubes, electronic and electrical equipment, and energy and chemical industries ^[1-4]. With the requirements of energy saving and environmental protection, copper tubes with thinner walls and smaller diameter have been attracting more and more attention. Therefore, it is essential to improve the mechanical properties and corrosion resistance of copper tubes to meet the demand. Microalloying can be an effective method to improve the strength and corrosion resistance of copper tubes ^[5,6].

Rare earth (RE) elements, known as the vitamins of industry, have been widely used as alloying additives to improve the properties of ferrous metals, aluminum alloys, magnesium alloys and copper alloys ^[7-10].

Generally, there are two ways for RE elements to impose on copper and copper alloys: purification and alloying ^[11-15]. Zhang et al. found that RE element Ce can remove the Fe, Cl, S, P and other impurities from pure copper, and when the Ce content reached 0.04%, pure copper obtained fine microstructure and favorable comprehensive mechanical properties: the tensile strength, yield strength, and elongation after annealing were 251 MPa, 116 MPa, and 37.22%, respectively ^[16]. According to Ref. ^[17], La had obvious effects on the solidification microstructure and the grain refinement of as-cast pure copper. With the increase of La content, the ultimate tensile strength, yield strength and microhardness increased gradually, but the elongation increased first and then decreased while La content exceeded 0.089wt.%. It suggested that the effects of grain refinement strengthening, solid solution strengthening, second phase strengthening and purifying by RE elements led to the improvement of mechanical properties. Except for pure copper, many studies focused on the effects of RE elements on copper alloys or composites ^[18-21]. For example, Mao et al. found that RE elements had an obvious effect on refining dendrite structure and grain size, as well as on purifying the melt of Cu-30Ni alloy. With the increase of RE content,

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the ultimate tensile strength, yield strength and elongation increased at first and then decreased after RE addition exceeded 0.095wt.%. The improvement of mechanical properties of Cu-30Ni-0.095RE alloy was attributed to microstructure refining and matrix purifying by RE^[22]. Wu et al. thought that RE could refine the as-cast microstructure and facilitate the precipitation of secondary Fe particles in the copper matrix by adding (0.05–0.30)wt.% rare earth elements into Cu-6wt.%Fe composites^[23]. Moreover, according to Ref.^[24–26], it was found that the galvanic corrosion was reduced, a dense corrosion product film was formed by the purification effect of rare earth, so the corrosion resistance of copper and copper alloys (i.e. imitation-gold copper alloy and Al-brass) with RE addition was improved significantly as compared with the initial alloy without RE addition.

According to the previous studies, adding small RE elements into copper and copper alloys would result in the improvement of the comprehensive properties of alloys. Most of the experiments were carried out under laboratory conditions or under vacuum conditions, however, the reports of RE applications in actual production processes are rather limited, especially in the studies applying RE in the HCC copper tube process. Therefore, in this paper, RE La was applied to the actual HCC copper tube process, and its effect on the microstructure,

mechanical and corrosion properties of HCC TP2 copper tube was evaluated systematically.

1 Experimental procedure

Experiments were carried out in the horizontal continuous casting system for TP2 copper tube, which consisted of melting furnace, crystallizer and dragger. The electrolysis copper plate was melted in the main (or utility) frequency induction furnace. At 1,150 °C, the Cu-La master alloys were added into the copper melt covered with charcoal and graphite flake. The La content in the master alloys was approximately 15wt.%. The addition content of La in copper melt was 180 ppm. The TP2 copper tube billet was drawn after the temperature of the melt reached 1,150 °C and held for 1 hour. The withdrawing speed of samples No.1 and 3 was 378 mm•min⁻¹, while that of sample No. 2 was 336 mm•min⁻¹.

The chemical compositions of the specimens were determined using a SPECTRO OES unit, and the La content in the TP2 copper tube was measured by an Optima 7300 DV ICP-OES. The specimens were tested at 22 °C and 50% humidity. The chemical compositions of the HCC TP2 copper tube billets are shown in Table 1.

Table 1: Chemical compositions of HCC TP2 copper tubes (ppm)

No.	Cu (%)	P	As	Fe	Pb	Zn	S	O	Si	Ag	La
1	99.977	182.7	5.0	19.7	1.0	5.5	4.4	58	1.0	6.3	0
2	99.975	197.7	5.0	19.5	1.0	5.4	1.0	13	1.0	6.3	27
3	99.975	197.1	5.0	19.6	1.0	5.3	1.0	11	1.0	6.3	30

The specimens cut from the HCC TP2 copper tube billets were polished with a series of abrasive papers and etched by 4% HNO₃ and 5% FeCl₃+5% HCl+90% C₂H₅OH solution before metallographic observation. The microstructures were observed by a ZMEF4A OM and a SSX-550 SEM equipped with EDS in secondary electron mode operated at 15 kV. The compositions of phases were examined by EDS. The grain sizes were measured by intercept method using an image-pro plus software. Tensile tests were performed on an INSTRON 5582 electronic universal testing machine at ambient temperature with a tensile rate of about 1 mm•min⁻¹ according to GB/T 228.1-2010 using rod sample and sheet sample. The samples are schematically shown in Fig. 1. The fractography characteristics were examined by SEM.

Fig. 1: Diagram of tensile test sample: (a) rod sample at axial direction, (b) sheet sample at transverse direction

The corrosion behavior in 0.5 mol•L⁻¹ HCl solution at room temperature of the TP2 copper tube with La microalloying was evaluated using a CS300 electrochemical workstation. The working electrode, reference electrode and counter electrode were the sample, a saturated calomel electrode (SCE) and a platinum electrode, respectively, in a standard three-electrode cell. In the present research, all the potentials were referred to SCE. The samples were mounted with an exposed area of 100 mm² using epoxy resins, and were polished to mirror-like finish before the corrosion test. The potentiodynamic polarization tests were performed at a scan rate of 0.3 mV•s⁻¹. In order to ensure the accuracy of the results, all electrochemical measurements were repeated three times.

2 Results and discussion

2.1 Absorptivity of La in TP2 copper tube

Table 2 shows the addition and absorptivity of La in HCC TP2 copper tube. Due to the active chemical properties of La, it is easy to react with other elements in copper and form clinkers, which led to the loss of La when La is added to the molten copper. Chen et al.^[27] reported that the limited absorptivity of La in copper under vacuum conditions was

Table 2: Absorptivity of La in TP2 copper tubes under antivacuum condition

No.	Addition (ppm)	Practical content (ppm)	Burning loss (%)	Absorptivity (%)
1	0	0	-	-
2	180	27	85	15
3	180	30	83.3	16.7

93%. The burning loss of La under antivacuum conditions is obviously higher than that under vacuum conditions. The burning mechanism under antivacuum conditions is similar to that under vacuum conditions. Furthermore, there are some impurity elements in the copper melt, such as O, H, S, Pb, Bi and Fe, and when La is added into the molten bath, it easily reacts with the impurities to form some compounds with high melting points and then be removed as slag. On the other hand, it is unavoidable that La reacts with oxygen in the air to form lanthanum oxide because of its high chemical activities. RE oxide with a high melting point and lower density is also removed to dross. Consequently, the remaining content of La in the HCC TP2 copper tube reduces greatly compared to its addition amount under antivacuum conditions.

Figure 2 and Table 3 show the EDS analysis results of casting dross. It can be seen that the casting dross contains O, P, S, Cl, Cu, La and other elements, which demonstrated that La deprives O, P, S, Cl and other impurities from the copper melt. The La, which influences the composition of the TP2 copper tube, has played a role in purifying the copper melt.

Fig. 2: EDS analysis of casting dross
Table 3: EDS analysis of casting dross

Element	mass%	at.%
O	23.701	63.721
P	5.942	8.252
S	1.342	1.801
Cl	2.502	3.036
Cu	7.065	4.782
La	59.448	18.408
Total	100	100

2.2 Microstructure

Figure 3 displays the solidification structures of HCC TP2 copper tubes with and without La addition. It can be seen that, whether or not the La was added, there is inhomogeneous dendritic distribution along the circumferential direction, i.e., the columnar dendrite on the bottom of the tube billet is coarser than that on the upper side of the tube. The slight difference between the upper side and the bottom of the tube billet is normal in the horizontal continuous casting. This is due to the non-uniform heat dissipation between the upper side and the bottom of the tube billets during the horizontal continuous casting^[28]. However, as shown in Fig. 4 and Fig. 5, the microstructure of HCC TP2 copper tubes with La addition was refined significantly. After La addition, the average columnar dendrite spacing was reduced to 0.93 mm with a reduction of approximately 57% compared with the average columnar dendrite spacing of 2.21 mm of the copper tube without La addition. The refinement mechanism can be described as follows: according to the metal solidification theory, when La was added to the molten copper, La element would be enriched at the front of the solid-liquid interface during the solidification process because of its lower melting point. Consequently, the constitutional undercooling degree at the front of the solid-liquid interface and the driving force of nucleation were increased during the solidification^[29]. Thus, the nucleation velocity was enhanced, and the grains were refined. On the other hand, La element easily reacted with some impurity elements in copper, such as O and S, to form oxide compounds, such as La_2O_3 , $\text{La}_2\text{O}_2\text{S}$ and La_2S_3 . According to Ref.^[30, 31], the melting points of La_2O_3 , $\text{La}_2\text{O}_2\text{S}$ and La_2S_3 are 2,249 °C, 1,940 °C and

Fig. 3: Solidification structures of TP2 copper tube with and without La addition: (a) 0 La, (b) 27 ppm La, (c) 30 ppm La

Fig. 4: Microstructures of TP2 copper tubes with and without La addition: (a) 0 La, (b) 27ppm La, (c) 30ppm La

Fig. 5: Average columnar dendrite spacing of TP2 copper tube with and without La addition: #1 0 La, #2 27 ppm La, #3 30 ppm La

2,099 °C, respectively. Thus, these compounds would solidify much earlier than copper during the solidification process. Because of that, these precipitated phases can provide enough nucleating sites, and the growth of the columnar grains is impeded. As a result, the grains of the HCC TP2 copper tube with La addition were refined.

2.3 Tensile properties

Table 4 shows the ultimate tensile strength and elongation of HCC TP2 copper tubes with and without La addition. It can be seen that the ultimate tensile strength in the axial direction is almost at the same level as that in the transverse direction; while the elongation in transverse direction is much higher than that in the axial direction because the tensile direction is consistent with the dendrite growth direction. It can also be seen from Table 4 that the ultimate tensile strength and the elongation in axial direction are almost kept at the same levels with or without La addition, while, they are slightly improved in transverse direction. Figure 6 gives the SEM fractographs of tensile samples of the TP2 copper tube before and after La addition. It is obvious that some dimples distribute in the fracture surfaces of all samples, indicating the microscopic characteristics of ductile fracture. Therefore, combined with results in Table 4, it can be concluded that trace La has a small effect on the ultimate tensile strength and the elongation of HCC TP2 copper tube.

Table 4: Results of tensile tests

Sample No.	Axial direction		Transverse direction	
	Tensile strength (MPa)	Elongation (%)	Tensile strength (MPa)	Elongation (%)
1	160(±1.2)	40.5(±0.5)	141(±1.1)	85.8(±0.5)
2	171(±0.2)	40.3(±1.2)	166(±1.6)	84.9(±1.0)
3	167(±1.5)	40.5(±0.8)	158(±1.0)	90.8(±1.2)

Fig. 6: SEM fractographs of tensile samples of TP2 copper tubes: (a) 0 La, (b) 27ppm La, (c) 30ppm La

2.4 Corrosion properties

Figure 7 shows the potentiodynamic polarization curves of HCC TP2 copper tubes before and after La addition in 0.5 mol·L⁻¹ HCl solution at 25 °C. Table 5 lists the electrochemical parameters obtained from the potentiodynamic polarization curves. Annual corrosion rate V_A can be calculated by Eq. (1):

$$V_A = \frac{3.16 \times 10^8 I_{\text{corr}} M}{z F \rho} \quad (1)$$

where, I_{corr} is corrosion current density, M is metal molar mass (g·mol⁻¹), z is electron transfer number of a single metal atom, F is Faraday constant (C·mol⁻¹), ρ is metal density (g·cm⁻³).

Fig. 7: Potentiodynamic polarization curves of different TP2 copper tubes in 0.5 mol·L⁻¹ HCl solution at 25 °C

From Fig. 7 and Table 5, it can be seen that the corrosion potential E_{corr} of the HCC TP2 copper tube with La addition is higher than that without La addition, while the corrosion current density I_{corr} and annual corrosion rate V_A of the HCC TP2 copper tube with La addition are lower than that without La addition. After La addition, the cathodic polarization curve of HCC TP2 copper tubes moves to the positive direction, the corrosion potential of copper tubes increases from -679.31 mV to -471.91 mV and -521.67 mV, respectively; and at the same time, the corrosion current density significantly decreases from 432.2 $\mu\text{A}\cdot\text{cm}^{-2}$ to 398.04 $\mu\text{A}\cdot\text{cm}^{-2}$ and 417.64 $\mu\text{A}\cdot\text{cm}^{-2}$, and the annual corrosion rate decreases from 10.18 to 9.84 and 9.37 mm·a⁻¹ correspondingly, suggesting that the corrosion resistance of HCC TP2 copper tubes is improved by virtue of La addition.

The higher the corrosion potential E_{corr} , the more difficult the electronic loss becomes, and the better the corrosion

Table 5: Electrochemical parameters obtained from potentiodynamic polarization curves of different TP2 copper tubes

Sample No.	1	2	3
$E_{\text{corr}}^*(\text{mV})$	-679.31	-471.91	-521.67
$I_{\text{corr}}(\mu\text{A}\cdot\text{cm}^{-2})$	432.2	417.64	398.04
$V_A(\text{mm}\cdot\text{a}^{-1})$	10.18	9.84	9.37

*: E_{corr} is corrosion potential

resistance of a material in thermodynamics. The lower the corrosion current density, the better the corrosion resistance of a material^[32]. Figure 7 shows that the shapes of the polarization curves before and after La addition are similar, indicating that the corrosion mechanisms are the same. The corrosion mechanisms are all controlled by cathode reactions, while the effect of electrochemical corrosion mechanism is weak. There are some impure elements in copper, such as O, H, S, Pb, Bi, and Fe. The impurity elements can form compounds or eutectic with copper, which have a potential higher than that of copper. The compounds or eutectic can act as cathode in the corrosive medium and copper acts as anode, so the existence of impure elements in copper promotes the electrochemical corrosion of copper. Due to its active chemical properties, La can react with the harmful impurity elements in copper. Therefore, the impurities can be removed by La addition. The areas of cathode were reduced, and then the cathode reaction was restrained, lowering the corrosion speed of anode copper. The galvanic corrosion was weakened and the corrosion resistance of HCC TP2 copper tubes was improved. As shown in Table 1, the content of impurity elements (O, S, et al) in copper are decreased, hence, the corrosion resistance of TP2 copper tube becomes better.

3 Conclusions

The effects of trace La microalloying on microstructures, mechanical and corrosion properties of HCC TP2 copper tubes were investigated. The following conclusions can be drawn based on the results of the experiment:

- (1) The absorptivity of La in HCC TP2 copper tube in the actual production conditions is about 15%, which is much lower than that under vacuum conditions.
- (2) The average columnar dendrite spacing of the HCC TP2 copper tube with La addition are refined obviously from 2.21 mm to 0.93 mm.
- (3) Trace La has a small effect on the ultimate tensile strength and the elongation of HCC TP2 copper tubes. However, the annual corrosion rate decreases from 10.18 mm·a⁻¹ to 9.37 mm·a⁻¹, so the corrosion resistance of the HCC TP2 copper tubes in HCl solution is improved by the purifying effect of La.

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