

Research on alloying the Cu-Ni-Sn system equivalent to grade C72500 in vacuum induction furnace

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ABSTRACT

In this article, research on smelting technology of elastomeric copper alloy of Cu-Ni-Sn system grade C72500 in vacuum induction furnace and some results of studying the structure and mechanical properties of C72500 alloy were shown. The C72500 alloy was smelted in a vacuum medium frequency furnace with a small burning rate and high cleanliness. Some methods to evaluate by determining structure, mechanical properties as: EDX spectroscopy analyzes element content, Optical microscope equipment to determine microscopic structure, ultrasonic equipment to evaluate defects and equipment to test mechanical properties. The after-casting alloy has a chemical composition and mechanical properties equivalent to imported copper alloys according to ASTM B122/B122M-20 standards, single α phase structure, good ductility but low strength, elongation 62.2%, tensile limit 301.43 MPa, yield limit 171 MPa and hardness distributed along the sample, high on the outside (82.4 HV) and low in the center (73.4 HV), is used in manufacturing elastic, abrasion and corrosion resistant parts.

Keywords: Cu-Ni-Sn; C72500 alloy; Vacuum induction furnace; Mechanical properties.

1. INTRODUCTION

Cu-Ni-Sn alloy has been widely used in the aerospace industry, electronics industry and other fields due to its excellent electrical and thermal conductivity, high strength, corrosion resistance and ability to anti-abrasion [1, 2], etc. Among copper alloys, Cu-Be alloy once played an indispensable role in the economic industry and was known as the "king of non-ferrous metal elastic materials" [3]. However, the production of Cu-Be alloys generates toxic dust and their stress relief performance and electrical conduction stability tend to degrade significantly at high temperatures, making it necessary to have a material system. New materials to solve and limitations of these problems in their applications [4]. With the rapid development of the electronics industry in the direction of miniaturization and highly reliable integration, the production of copper alloys with high durability, elasticity, safety, non-toxicity, and simplicity is essential. Simple to prepare, saving costs is very important. A series of copper-based and beryllium-free elastomeric alloys have been successfully developed, such as Cu-Ti [5], Cu-Ni-Sn [6], Cu-Ni-Mn [7], Cu-Ni-Al [8] and Cu-Fe-P [9]. In the 1970s, Bell Telephone Laboratories successfully created Cu-Ni-Sn alloy and it was widely developed [10]. The main Cu-Ni-Sn alloys are Cu-9Ni-2Sn (UNS C72500), Cu-4Ni-4Sn (UNS C72600), Cu-9Ni-6Sn (UNS C72700), Cu-10Ni-8Sn (UNS C72800) and Cu-15Ni-8Sn (UNS C72900). The mechanical properties and wear resistance of Cu-Ni-Sn alloy are similar to those of Cu-Be alloy, while the thermomechanical workability and corrosion resistance of Cu-Ni-Sn alloy are better than Cu-Be alloy [11].

We can determine the phase structure and alloying ability of the Cu-Ni-Sn alloy system, thereby explaining the material selection processes, smelting technology and heat treatment to create the desired properties of the material. However, there are still many problems that need to be solved in the research of smelting Cu-Ni-Sn system materials such as: There will be large loss when smelting in an environment without protective gas, bias in composition, need to study more

deeply the relationship between heat treatment process and structure and properties of the alloy, improve the ductility, electrical conductivity and thermal conductivity of alloy and exploit the maximum potential of alloy, prevent Sn segregation and apply them to actual production as soon as possible to improve alloy properties, etc. The content of this paper focuses on the study of alloying of Cu-Ni-Sn system equivalent to grade C72500 (according to ASTM B122/B122M-20 standard) in a vacuum medium frequency induction furnace to study the alloying smelting technology and evaluate the products after smelting.

2. EXPERIMENTAL

2.1. Materials and equipment

Material: Pure copper (99.99%), Nickel (99.99%), Tin (99.99%); Vacuum Induction Furnace - VIF; Tensile testing equipment WDW-100Y; Electron microscope SU1510; Phased array ultrasonic flaw detector VEO+16:64PR; Optical microscope equipment Axio CSM 700; Equipment X-ray Bruker D8-advance.

2.2. Smelting process of C72500 alloy in vacuum induction furnace

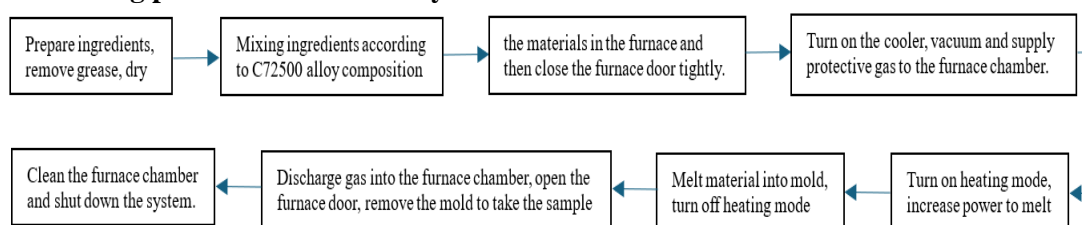


Figure 1. Sequence diagram of the smelting process steps in a vacuum induction furnace.

3. RESULTS AND DISCUSSION

3.1. Chemical composition of C72500 alloy after casting

C72500 copper alloy is smelted in a vacuum induction furnace. Based on the chemical composition of C72500 alloy according to ASTM B122/B122M-20 standard in table 1, the article used input materials and chemical composition analysis results by emission spectroscopy at Quastest Center 1 (table 2) to calculate the loss coefficient of alloy elements (Cu, Ni, Sn) when smelting in a vacuum furnace in table 3.

Table 1. Chemical composition of C72500 alloy [12].

Alloy grade	Ni	Sn	Fe, max	Mn, max	Zn, max	Pb, max	Cu
C72500	8.5-10.5	1.8-2.8	0.6	0.2	0.5	0.05	Remaining

Table 2. Chemical composition through smelting batches.

Casting batch	Ni	Sn	Fe	Mn	Zn	Pb	Cu
1	10.7972	2.8755	0.0158	0.0127	0.0053	0.0136	86.26
2	8.4972	2.9078	0.014	0.0135	< 0.0005	0.0075	88.54
3	8.5244	2.7834	0.0151	0.0145	0.0005	0.0061	88.61
4	8.6281	2.1554	0.0143	0.0106	0.0005	0.0046	89.17

In table 2, the results show that the casting 1 and 2 of the main alloy elements Ni and Sn have not met the composition requirements. In the experiment, casts 3 and 4 both met the chemical composition requirements of C72500 alloy grade, equivalent to the same alloy grade in the American standard ASTM B122/B122M-20. However, cast 4 limited the nickel and tin content to a better level within the allowable range of the standard, so the research results took samples of cast 4 to make the next research sample. The content of elements is within the allowable limit, impurities such as Pb, Fe, Mn, Zn all have very low content ensuring the alloy has high purity.

Through the calculation results of the loss of Cu, Ni, Sn elements in table 3, they are all very small (less than 3%) compared to when smelting in an environment without protective gas (less than 10%).

Table 3. Mixtures for casting C72500 alloy.

Material	Cast 1 (g,%,%)			Cast 2 (g,%,%)			Cast 3 (g,%,%)			Cast 4 (g,%,%)		
	Mix	Anal	Lost	Mix	Anal	Lost	Mix	Anal	Lost	Mix	Anal	Lost
Cu	444	86.26	1.28	455.5	88.54	1.17	456	88.61	1.26	458.8	89.17	1.08
Ni	55	10.79	0.25	43.3	8.497	0.23	43.5	8.524	0.42	44	8.628	0.19
Sn	15	2.876	2.59	15.2	2.908	2.74	14.5	2.783	2.46	11.2	2.155	2.05
Initial mass, g	514			514			514			514		
Weight after casting, g	508.12			508.42			508.14			508.96		

The chemical composition of the C72500 alloy at cast 4 was analyzed by EDX spectroscopy and the results are shown in Fig. 2. The contents of Cu, Ni, Sn elements are 87.58%, 9.82% and 2.6% in Fig. 2(a) and 87.22%, 9.85% and 2.93% in Fig. 2(b) respectively.

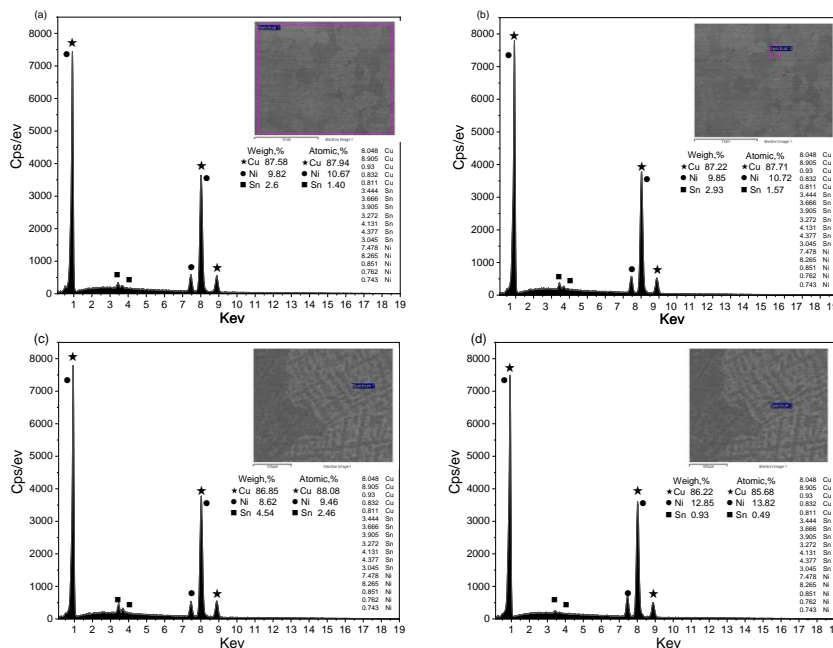


Figure 2. EDX compositional analysis results of the alloy after casting:

(a) Wide region; (b) Narrow region; (c) White point, (d) Black point.

In the white area, the Sn content is higher than in the dark area, in the process of dendritic crystallization, the rate is higher than the concentration diffusion process. The EDX analysis results at the points on the dendrite are the white point in Fig. 2 (c) and the black point in Fig. 2 (d). The results show that the black point is the background with higher Cu and Ni content than the white point. The white point on the dendrite has a higher Sn content of 4.54% compared to 0.93%. The reason is that during the crystallization process of metal changing from liquid to solid phase, the elements that crystallize first to form solid particles have a higher content than the solid phase components in the particles that crystallize later. The ratio of each chemical component is basically the same as in the initial mixture before smelting, consistent with the smelting process results, the diffusion crystallization of the component when cooling, meeting the composition requirements of the C72500 alloy.

3.2. Microstructure and phase composition of C72500 alloy

Metal molds are used to cast copper alloys, due to their high thermal conductivity and heat dissipation capacity, fast cooling speed, compact casting structure, and good casting mechanical properties. In addition, metal molds can be reused many times. The microstructure of the as-cast C72500 alloy is shown in Fig. 3. The microstructure of copper alloys during cooling is the crystallization process of solid solution is the process of nucleation and growth of nuclei. The nucleation of a solid solution requires structural and energy fluctuations. In addition, since the solid phase composition crystallized by the solid solution is different from that of the original liquid solution.

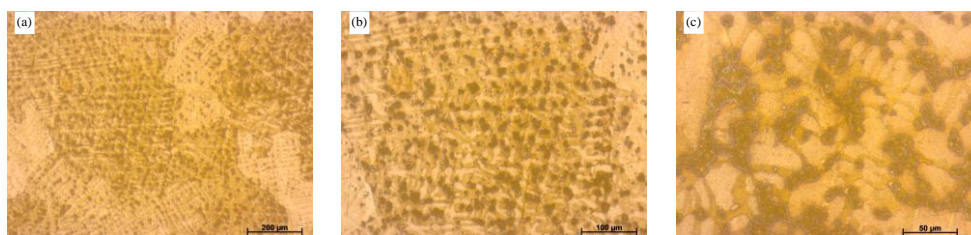


Figure 3. Microstructure of C72500 alloy after casting at different magnifications. (a) x50; (b) x100; (c) x200.

Fig. 3 (a) shows that the solid-state alloy structure consists of large-sized particles, within which is a dendritic structure formed during crystallization as shown in Fig. 3 (b) and Fig. 3 (c). The cooling rate and diffusion process are insufficient under certain conditions, which causes the solid-liquid phase to maintain a certain concentration gradient, resulting in uneven composition in the phase. The microstructure of the α single-phase solid solution is in the form of a dendrite. When cast in a metal mold, due to the rapid cooling rate, the crystallization rate is greater than the diffusion rate of the components, so the resulting structure is in the form of a dendrite and the particles have uneven composition. This is clearly shown when analyzing the composition by EDX method between particles and the uneven composition at the dendrites in the same particle as in Fig. 3 (c) and Fig. 3 (d). Microsegregation occurs due to selective crystallization of the component with high crystallization temperature crystallizing first and the component with low crystallization temperature crystallizing later, and limited diffusion rate. In addition, there is also heterogeneity between regions due to the phenomenon of weight segregation crystallization when the heavy component sinks and the light component floats, this is also known as the phenomenon of macrosegregation. The grain size after casting is large, the grain size is coarse and very heterogeneous, so it is not beneficial for the mechanical processing in the next steps.

In Fig. 4, the X-ray diffraction pattern of C72500 alloy after smelting with a scanning angle from 30° to 80° , compared with the pure copper sample, it can be seen that in the cast alloy there is only one α phase, at the characteristic peaks 43.61° , 50.64° , 74.52° corresponding to the peaks of copper corresponding to the (111); (200); (220) faces with the lattice constant of Cu being 3.615 \AA [13].

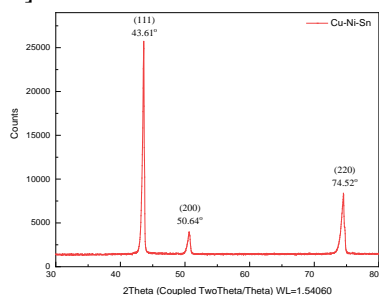


Figure 4. X-ray of C72500 alloy after casting.



Figure 5. Ultrasonic defect inspection.

Ultrasonic analysis results of defect testing of alloy samples after casting were performed at Z183 factory (Fig. 5). The analysis results showed that no micro-cracks, pitting, or delamination were detected on the C72500 copper alloy casting sample after smelting. This once again confirmed that the alloy after smelting met the chemical composition requirements according to the standard and that no defects appeared in the structure of the alloy, ensuring the mechanical properties of the alloy for further studies.

3.3. Mechanical properties of C72500 alloy

A typical room temperature tensile strength curve of C72500 alloy is shown in Fig. 7.

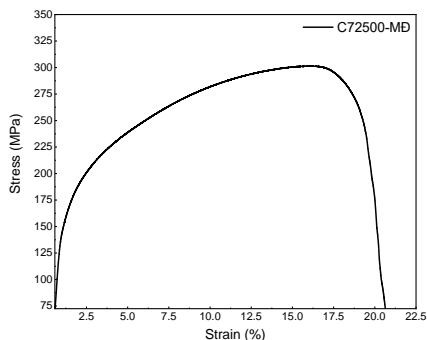


Figure 6. Tensile curve of C72500 alloy after casting.

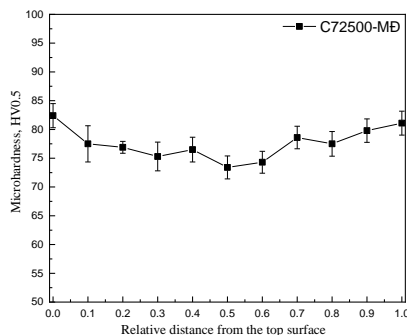


Figure 7. Graph of hardness distribution of C72500 alloy after casting.

In Fig. 6, the average yield strength of the cast alloy is only 171 MPa, the average tensile strength is 301.43 MPa and the average elongation is 62.2%. The microstructure of the C72500 alloy consists of coarse grains and heterogeneous distribution, which is the reason for the low strength of the cast alloy. The hardness distribution of the alloy after casting was measured on a HV microhardness tester with a load of 0.5 kg, the measurement results are shown in Fig. 7. The results of microhardness measurement HV0.5 show that the average hardness of the alloy distributed from the outside to the core of the casting has changed, on the surface of the casting the hardness (82.4 HV) is higher than in the core (73.4 HV), this can be explained because when the casting cools, the outside of the casting contacts the metal mold so the speed is fast, so the crystallization process of the obtained grains is finer than the inside, so the hardness is higher. To suit the subsequent uses of this alloy, it is necessary to conduct heat treatment and deformation machining.

4. CONCLUSIONS

In this study, Cu-Ni-Sn alloy system was mixed in a vacuum induction furnace. After smelting, the resulting alloy had high purity, very low impurity content, chemical composition and mechanical properties equivalent to imported copper alloy grade C72500 according to ASTM B122/B122M-20 standard. The elements Cu, Ni, Sn with small loss rate (less than 3%). The results of EDX spectrum analysis showed that the content of Cu, Ni, Sn elements when analyzing the regions and points were different, the content of Cu, Ni, Sn elements in the wide region and narrow region were 87.58%, 9.82%, 2.6% and 87.22%, 9.85%, 2.93%, respectively, the black point is the background with higher Cu and Ni content than the white point, the white point on the tree branch with higher Sn content is 4.54% compared to 0.93%. The structure of C72500 alloy cast by metal mold is a dendritic structure with coarse grain, average grain size over 500 μm , different size distribution and heterogeneous chemical composition on the dendritic structure, the results of ultrasonic defect analysis showed that without cracks, pitting, or delamination were detected on the casting sample. Mechanical properties of C72500 alloy after casting have good ductility but low strength, elongation is 62.2%, tensile strength is 301.43 MPa, yield strength is 171 MPa. The average hardness distributed along the sample is high on the

outside (84.2 HV) and low in the center of the sample (73.4 HV). XRD diffraction analysis results show that the structure formed after casting is a single-phase structure (α phase) with lattice parameters of alloyed copper.

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REFERENCES

- [1]. Kim, Y.-K., S.-H. Park, and K.-A. Lee, "Effect of post-heat treatment on the thermophysical and compressive mechanical properties of Cu-Ni-Sn alloy manufactured by selective laser melting". *Materials Characterization*. 162: p. 110194, (2020).
- [2]. Guo, L., et al., "A review of Cu-Ni-Sn alloys: processing, microstructure, properties, and developing trends. *Materials*". 16(1): p. 444, (2023).
- [3]. Chen, L.-p. and Q. Zhou, "Research and application of beryllium copper alloy". *Mater & Heat Treat*. 38(22): p. 14, (2009).
- [4]. Shi, Y., et al., "Recrystallization behavior and mechanical properties of a Cu-15Ni-8Sn (P) alloy during prior deformation and aging treatment". *Materials Science and Engineering: A*. 826: p. 142025, (2021).
- [5]. Hanoz, D., et al., "Effect of Precipitation Hardening on Corrosion Resistance of Cu-4.5 wt.% Ti". *Journal of Materials Engineering and Performance*. 30: p. 1306-1317, (2021).
- [6]. Jiang, Y., et al., "Microstructure and properties of a Cu-Ni-Sn alloy treated by two-stage thermomechanical processing". *Jom*. 71: p. 2734-2741, (2019).
- [7]. Xie, G.L., et al. "Precipitation process and mechanical properties of an elastic Cu-Ni-Mn Alloy". In *Materials Science Forum*. 2015. Trans Tech Publ.
- [8]. Li, D.-y., et al., "Superelasticity of Cu-Ni-Al shape-memory fibers prepared by melt extraction technique". *International Journal of Minerals, Metallurgy, and Materials*. 23: p. 928-933, (2016).
- [9]. Dong, Q., et al., "Effect of thermomechanical processing on the microstructure and properties of a Cu-Fe-P alloy". *Journal of Materials Engineering and Performance*. 24: p. 1531-1539, (2015).
- [10]. Schwartz, L. and J. Plewes, "Spinodal decomposition in Cu-9wt% Ni-6wt% Sn-II. A critical examination of mechanical strength of spinodal alloys". *Acta Metallurgica*. 22(7): p. 911-921, (1974).
- [11]. Shankar, K.V. and R. Sellamuthu, "Determination of the effect of nickel content on hardness, optimum aging temperature and aging time for spinodal bronze alloys cast in metal mould". *Applied Mechanics and Materials*. 813: p. 597-602, (2015).
- [12]. 齐亮 and 柳瑞清, "铸态 C72500 合金组织与性能分析. *铜业工程*", (4): p. 49-51, (2005).
- [13]. Theivasanthi, T. and M. Alagar, "X-ray diffraction studies of copper nanopowder". arXiv preprint arXiv:1003.6068, (2010).

TÓM TẮT

Nghiên cứu hợp kim hóa hệ Cu-Ni-Sn tương đương mác C72500 trong lò cảm ứng chân không

Trong bài báo này, nghiên cứu công nghệ nấu luyện hợp kim đồng đàn hồi hệ Cu-Ni-Sn mác C72500 trong lò cảm ứng chân không và một số kết quả nghiên cứu tổ chức và cơ tính của hợp kim C72500 sau khi nấu luyện. Hợp kim C72500 được nấu luyện trong lò cảm ứng chân không có mức độ cháy hao nhỏ, độ sạch cao. Các thiết bị nghiên cứu đánh giá tổ chức, tính chất hợp kim như: Quang phổ EDX phân tích hàm lượng nguyên tố, thiết bị hiển vi quang học xác định tổ chức tế vi, thiết bị siêu âm đánh giá khuyết tật, thiết bị kiểm tra độ bền cơ tính. Hợp kim sau đúc có thành phần hóa học và cơ tính tương đương với mác hợp kim đồng nhập ngoại theo tiêu chuẩn ASTM B122/B122M-20, tổ chức một pha α , có độ dẻo tốt nhưng độ bền thấp, độ giãn dài 62,2%, giới hạn bền kéo 301,43 MPa, giới hạn chảy 171 MPa và độ cứng phân bố dọc theo mẫu cao ở ngoài (82,4 HV) và thấp ở trong tâm (73,5 HV) được ứng dụng trong chế tạo các chi tiết đàn hồi, chịu mài mòn và ăn mòn.

Từ khóa: Cu-Ni-Sn; Hợp kim C72500; Lò cảm ứng chân không; Tính chất cơ lý.