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Microstructural and Hardness Studies of Cu-10wt.%Sn Alloy Under Different Aging Conditions

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Microstructure of Cu-10wt.%Sn alloy, prepared by powder metallurgy technique and sintered at 900 °C for 120 min in hydrogen atmosphere, was studied by optical microscopy and XRD technique as a function of aging time. Isothermal aging of the alloy specimens was performed at 250 °C for a period of 30, 60, 120, 300, and 1440 min after solution treatment at 500 °C for 60 min. Rockwell hardness of aged specimens was also measured at room temperature as a function of aging time. It was observed that microstructure of the as-sintered specimens consists of the grains of alpha Cu-Sn solid solution. Moreover, solution treatment of the alloy specimens followed by quenching in water increased the hardness of the as-sintered alloy specimens from 35.5 to 59.8 HRF due to the residual stresses generated by fast cooling. Aging at 250 °C for 30, 60, and 120 min was found to cause a decrease in hardness from 59.8 to 45.1 HRF, whereas the specimens aged for 300 and 1440 min show an increase in hardness from 45.1 to 75.7 HRF. The values of porosity calculated from XRD patterns of the alloy specimens referred to show that porosity varies with aging time in a manner opposite to that of hardness, e.g., porosity is maximum for 120 min aging time where hardness is minimum.

Keywords aging, bronze, hardness, microstructure, porosity

1. Introduction

The copper-tin alloys containing 8-12 wt.% tin are used for gears and other machine parts for self-lubricated bearings that are heavily loaded, for filters that are porous, and for marine fittings to resist impingement attack by seawater. Extensive investigations have been made in the past to explore, e.g., the role of conventional and microwave modes of sintering, dilation effects during sintering, and difference between various properties of these alloys produced by powder metallurgy technique and in the molten state. For instance, Monaghan et al. (Ref 1) studied thermal properties, namely heat capacity, thermal diffusivity, and electrical resistivity of a Cu-10wt.%Sn alloy in both solid and liquid phases.

Acharya (Ref 2) carried out thermal analysis of slow-cooled copper-tin alloys over the entire composition range to investigate and compile dilation effects accompanying various phase reactions that occur in the compacts as they are heated during sintering. Sethi et al. (Ref 3) studied the sintering behavior of Cu-10wt.%Sn alloy in both microwave furnace as well as conventional furnace, and found that this alloy was sintered by microwave heating in significantly less time as compared to conventional sintering. Hardness of the premixed microwave specimens was also higher than that for the corresponding conventional premixed specimens, and microstructure in the case of former was rather more uniform. Therefore, for fabricating bearings and filters with higher porosity, they recommended to compact the premixed bronze at higher pressure followed by microwave sintering.

Hong et al. (Ref 4) investigated in detail acoustic characteristics of the bells made with Cu-Sn alloys for a range of Sn concentrations by using FFT type power spectrum analyzer. They found that the values of frequency and tonal intensity of the bell decreased with the increase in Sn concentration from 5 to 11 wt.% but thereafter magnitude of both the parameters referred to increased with further increase of Sn concentration. For a given Sn concentration, frequency and tonal intensity of the bell were also found to depend considerably on the porosity. Higher the porosity, lower were the values of frequency and tonal intensity of the bell made with a Cu-Sn alloy of a given composition.

The main object of the present work was to study microstructure and hardness of Cu-10wt.%Sn alloy prepared by powder metallurgy technique and then aged at a given temperature for different aging times. To explore relationship, if any, between hardness and porosity in this alloy was another objective.

2. Experimental Procedure

Appropriate amounts of 99.9% copper and 99.9% tin, both in the form of fine powder, were mixed and blended using citric acid as a lubricant. The mixture was compacted into a cylindrical shape (16 mm diameter and 30 mm height) using a stainless steel die under a pressure of 14 MPa (≈2000 psi) and was then sintered in a batch type electric furnace maintained at 900 °C for 120 min in hydrogen atmosphere.

The sintered material was cut into 3 mm thick 10 mm × 10 mm square pieces for various types of heat-treatment operations. In order to avoid oxidation of the

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material, specimens were sealed in evacuated glass tubes and were homogenized at 500 °C for 60 min inside an electric tube furnace. These were then quenched into iced water to form a supersaturated Cu-Sn solid solution. Each solution-treated specimen was again encapsulated in a glass tube under vacuum, and such tubes were placed inside the furnace maintained at 250 °C for isothermal aging for 30, 60, 120, 300, and 1440 min. During this aging treatment, tubes were taken out of the furnace one by one after the lapse of required aging times referred to above, followed by quenching of these aged specimens in iced-water to freeze the microstructure developed during aging.

Finally the specimens were polished to obtain a mirror like surface, and etched in a solution of acetic acid (50%) and nitric acid (50%) to reveal the microstructure. Metallographs shown in Fig. 1 were taken by Epimet Metallurgical Microscope N334, equipped with 135 mm camera CENTON DF 300 at a magnification of 200×. To detect the presence of any possible phase other than α-Cu (FCC), x-ray diffraction patterns were recorded at room temperature using Rigaku XRD C1-01 Diffractometer equipped with Cu Kα radiation using wavelength 1.5406 Å, tube voltage 20 kV, tube current 36 mA, and 2θ range from 20° to 80°. All the peaks in the diffraction patterns for sintered, solution-treated, and aged specimens were indexed for 2θ and *d*-values (Ref 5-7). These peaks were found to correspond to α-Cu (FCC) phase only in all the cases, like ones exemplified in Fig. 2, meaning thereby that no new phase, e.g., ε-Cu_3Sn, was developed in the specimens during aging carried out at 250 °C up to 1440 min. Rockwell hardness of all the Cu-10wt.%Sn alloy specimens was measured on HRF scale using Hardness Tester FR-1 Japan. Porosity was also calculated with the help of the XRD patterns by using the formula:

$$\text{Porosity}\,(\%) = [1 - (\rho/\rho_x)] \times 100 \qquad \text{(Eq 1)}$$

Here ρ is the measured mass density (i.e., mass/volume), $\rho_x = (n\bar{A}/NV)$ is the x-ray density, n is the number of atoms contained in the unit cell of volume V, i.e., 4 in case of alpha Cu-Sn (FCC), N is Avogadro's number, and $\bar{A}$ is the mean atomic weight of the atoms.

3. Results and Discussion

Figure 1 depicts microstructure of Cu-10wt.%Sn alloy specimens at a magnification of 200× for various stages of heat treatment: (a) sintered at 900 °C for 120 min, (b) solution-treated at 500 °C for 60 min, and then aged at 250 °C for (c) 30 min, (d) 120 min, (e) 300 min, and (f) 1440 min. The XRD patterns for solution-treated Cu-10wt.%Sn alloy specimens are shown in Fig. 2 together with some of the aged ones for comparison.

Now Fig. 3 illustrates the dependence of hardness of solution-treated as well as aged Cu-10wt.%Sn alloy specimens on aging time. Each point denotes an average value of five measurements for a given aging time. It can be seen that hardness initially decreases from 59.8 to 45.1 HRF with the

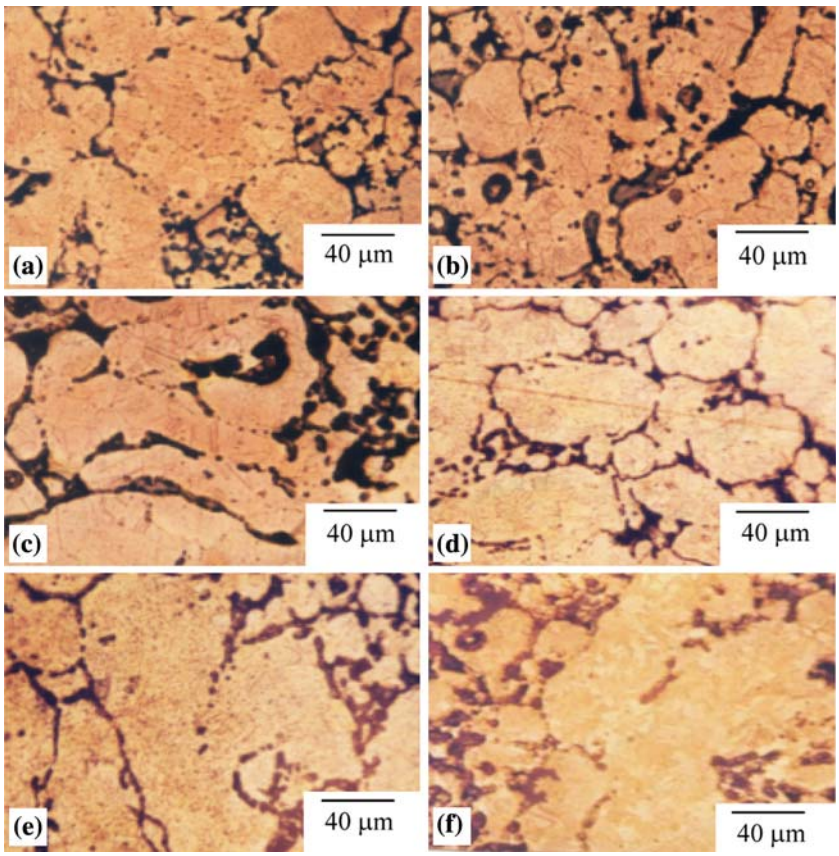

Fig. 1 Microstructure of Cu-10wt.%Sn alloy specimens: (a) sintered at 900 °C for 120 min, (b) solution-treated at 500 °C for 60 min, (c) aged at 250 °C for 30 min, (d) aged at 250 °C for 120 min, (e) aged at 250 °C for 300 min, and (f) aged at 250 °C for 1440 min

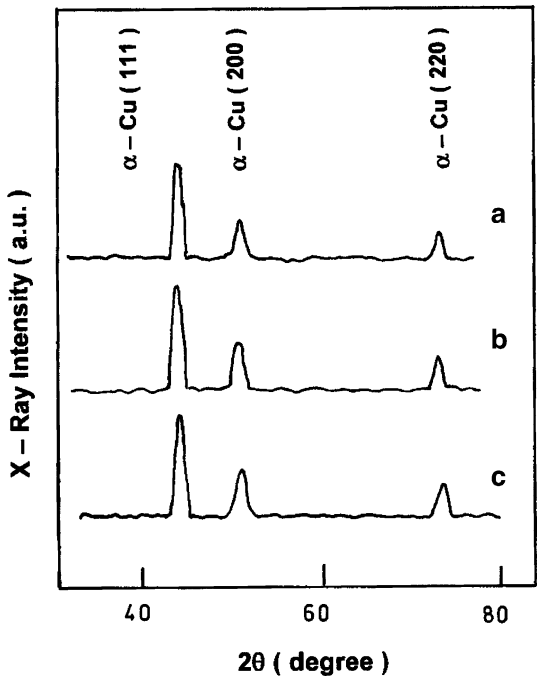

Fig. 2 The XRD patterns of Cu-10wt.%Sn alloy specimens: (a) solution-treated at 500 °C for 60 min, (b) aged at 250 °C for 30 min, and (c) aged at 250 °C for 1440 min

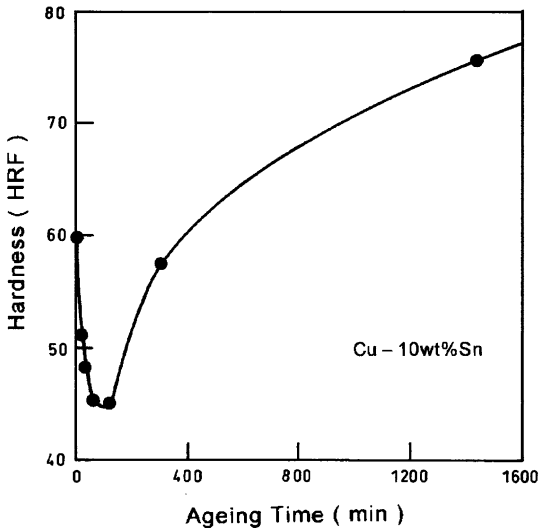

Fig. 3 Dependence of Rockwell hardness of Cu-10wt.%Sn alloy on aging time

increase in aging time till 120 min, and later on increases to 75.7 HRF with further increase in aging time to 1440 min. It should also be noted that hardness of as-sintered specimen was found to be 35.5 HRF while that of solution-treated and then quenched specimen was 59.8 HRF. This 68% increase in hardness may be attributed to the residual stresses generated by fast cooling. The initial decrease in hardness from 59.8 to 45.1 HRF during aging time interval 0-120 min may be a consequence of the removal of residual stresses generated by quenching, increase in grain size, and variation in concentration of pores.

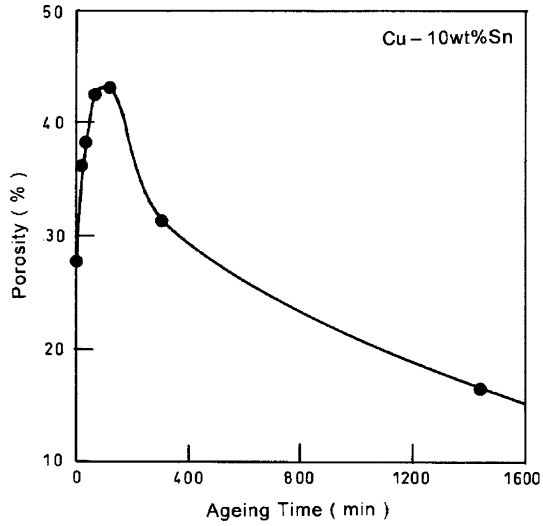

Fig. 4 Dependence of porosity in Cu-10wt.%Sn alloy on aging time

Similarly Fig. 4 depicts the variation of porosity in solution-treated as well as aged Cu-10wt.%Sn specimens as a function of aging time. Each point denotes an average value of five measurements for a given aging time. One can readily note that porosity first increases from 27.8% to 43.2% as aging time is increased from 0 to 120 min, and then decreases to 16.5% with the increase in aging time to 1440 min. It is worthy of mention that porosity in as-sintered specimen was found to be 30.9% while that of solution-treated and then quenched specimen was 27.8%. This 10% reduction in porosity may be attributed to the rearrangement of atoms during solution treatment at 500 °C leading to the reduction in porosity. The initial increase in porosity from 27.8% to 43.2% in the aging time interval 0-120 min can be a consequence of thermal agitation of atoms. However, prolonged aging times ($t > 120$ min) facilitate the observed reduction in porosity from 43.2% to 16.5% due to increase in grain size (Fig. 1). The *H-t* and *P-t* data given in Fig. 3 and 4 have been plotted in semi-logarithmic coordinates in Fig. 5 and 6, respectively. The lines drawn through the data points in each time interval, i.e., $t < 100$ min and $t > 100$ min, were found to be encompassed by the mathematical expressions:

$$H = A + B \ln t \quad \text{(Eq 2)}$$

and

$$P = C + D \ln t \quad \text{(Eq 3)}$$

The values of constants A, B, C, and D, along with correlation factor r in both the aging time intervals, i.e., 15-100 min and 120-1440 min, are given in Table 1. This shows that H and P vary exponentially with aging time t in a given interval of time.

Finally points in Fig. 7 depict the values of hardness as a function of porosity for all the Cu-10wt.%Sn alloy specimens which had undergone solution treatment at 500 °C for 60 min followed by quenching, and then aging at 250 °C for 15-1440 min. A straight line drawn through the data points was obtained by least-squares fit method, and is encompassed by the relation:

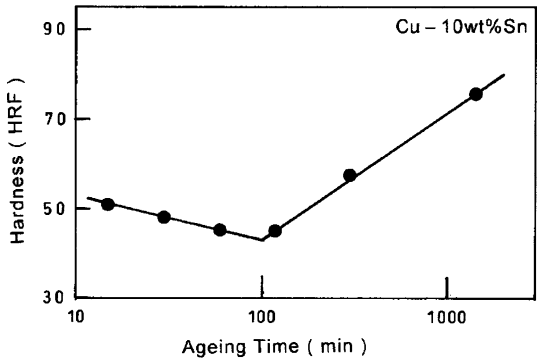

Fig. 5 Relation between Rockwell hardness of Cu-10wt.%Sn alloy and aging time in semi-logarithmic representation. Data points were taken from Fig. 3

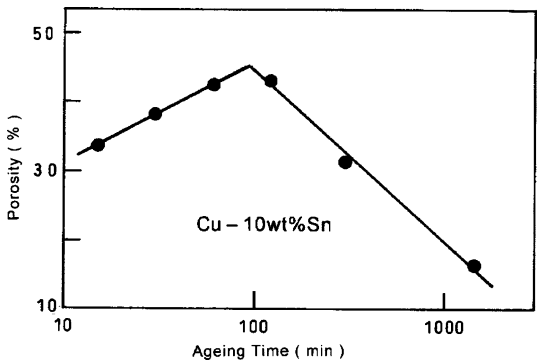

Fig. 6 Relation between porosity in Cu-10wt.%Sn alloy and aging time in semi-logarithmic representation. Data points were taken from Fig. 4

Table 1 Values of constants *A*, *B*, *C*, and *D* in Eq 2 and 3, along with that of correlation factor *r*

t (min)	*A* (HRF)	*B*	*r*	*C* (%)	*D*	*r*
15, 30, 60	+62.5	–4.2	–0.999	+17.2	+6.2	+0.999
120, 300, 1440	–13.0	+12.2	+0.999	+93.1	–10.6	–0.996

$$H = 93.31 - 1.14P \quad \text{(Eq 4)}$$

with a correlation factor $r = -0.994$. This shows that hardness strongly depends on the magnitude of porosity in the single-phase Cu-10wt.%Sn (FCC) alloy specimens and is independent of the thermal history when no second phase, e.g., ε-Cu_3Sn (HCP), exists in the original phase matrix. Lower the pore concentration, higher will be the hardness.

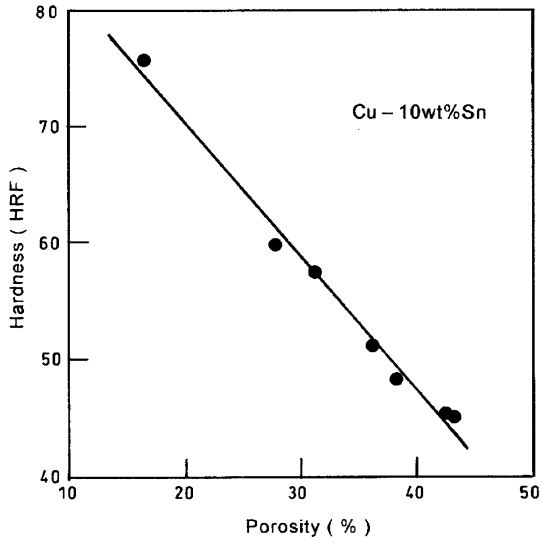

Fig. 7 Dependence of Rockwell hardness on porosity in the case of Cu-10wt.%Sn alloy

4. Conclusions

One may conclude from the foregoing evidence that the alpha phase Cu-10wt.%Sn solid solution, aged at 250 °C for 15-1440 min after solution treatment at 500 °C for 60 min, is very much stable and does not transform easily into ε-Cu_3Sn hexagonal phase. The Rockwell hardness of this single-phase alloy prepared by powder metallurgy correlates very well with the concentration of pores through the linear relationship $H = 93.31 - 1.14P$, with a correlation factor $r = -0.994$.

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