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PHYSICO-CHEMICAL CHARACTERISTICS OF THE Cu-Fe-P ALLOY WITH EMPHASIS ON THE DIFFRACTION ANALYSIS

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Abstract

This paper details the the analysis of a Cu-Fe-P alloy, containing 0.003 wt% Fe and 0.014 wt% P, conducted using the X-ray diffraction (XRD) and selected area electron diffraction (SAED) techniques. The melted and homogenized Cu-Fe-P alloy was subjected to the extrusion and cold working (rolling) with different degrees of deformation. The required mechanical and structural characteristics of material were achieved by the mechanical and thermal processing of the alloy, where the working conditions of extrusion corresponded to the rolling with 75% deformation, recrystallization annealing at 470℃, annealing time of 150 min and cooling in a mixture of water and alcohol in the ratio 3:1. Both XRD and SAED results confirmed the presence of a crystalline structure. The observed reflections matched the cubic structure of the Cu phase, with no reflections detected for Fe, P, or their compounds, which is likely due to the low concentrations of these alloying elements.

Keywords*: Cu-Fe-P, synthesis, characterization, XRD, TEM, SAED*

1. INTRODUCTION

Copper and its alloys are known for their exceptional mechanical properties, as well as their thermal and electrical conductivity. As a result, the demand for their production, processing, and application across various industries has significantly increased in recent years. Due to this reason, the other elements (Fe, P, Mo, Pb, Co, Ti, W, Cr, Zr, Ni, Si, etc.) were added to pure Cu, in order to obtain the new and appropriate properties of Cu [1-3].

The Cu-Fe alloys can be obtained using different methods. For instance, Zou et al. achieved a composite Cu-14Fe employing the casting with an alternating magnetic field [1], while Wang et al. developed the Cu-10 wt% Fe alloy using a combination of double melt mixed casting and multi-stage thermomechanical treatment [2]. Additionally, Ding et al. obtained Cu-Fe composite by the vacuum casting [3], and Tian et al. employed the powder rolling method [4]. Yang et al. studied the strength and electrical conductivity achieved by optimization the dual-phase structure in the Cu-Fe wires [5]. The authors analyzed the effect of microstructural parameters on the mechanical properties and electrical conductivity of wires. The result of wire drawing deformation significantly improved the strength of wires, while the electrical conductivity was not significantly reduced [5].

One of the important structural characteristics is the crushing of copper grains and copper alloys [6,7]. Thus, Feng et al. investigated the effect of casting and crystallization temperature on crushing the pure copper grains hardened by means of a pulsed magnetic field at low pressures [8]. They found that the higher temperatures led to the slower cooling rates, which allowed more time to mitigate the effects of pulsating magnetic field and thus achieved better grain refinement [8]. Lee et al. investigated the hierarchical microstructure and strengthening mechanisms of the Cu-36.8Fe alloy, produced by the selective laser melting [9]. Additionally, a common method for producing the Cu-Fe alloys involves using the metal powders [10-12].

In addition to synthesizing the Cu-Fe-P alloy, it is crucial to characterize it to confirm the properties, observed during the synthesis process. Accurate characterization ensures that the synthesized material meets the desired specifications and helps validate the effectiveness of the synthesis methods used. This study focuses on diffraction analysis, employing two distinct techniques to thoroughly investigate the structural characteristics of the alloy. By comparison the results, obtained from these techniques, it is aimed to provide a comprehensive understanding of the alloy composition, phase distribution, and crystallographic properties.

2. EXPERIMENTAL

To produce the Cu-Fe-P alloy, cathodic copper with 99.99% purity (GRADE A quality from AURUBIS) was utilized. The copper cathodes were melted in an electrical resistance furnace under a protective atmosphere. Following this, the alloy was cast using the "up-cast" process. Throughout the casting process, the chemical composition of the alloy was monitored continuously by sampling every hour. The contents of iron (Fe) and phosphorus (P) were measured using an optical emission spark spectrometer (OES), model 4460, manufactured by the ARL.

The X-ray diffraction (XRD) analysis was conducted using a Rigaku MiniFlex 600 equipped with a D/teX Ultra 250 high-speed detector and a Cu anode X-ray tube. The measurement covered a range of 3-90° with a step size of 0.02°, a recording speed of 10°/min, and operated at 40 kV and 15 mA. Crystalline phases were identified using the PDXL 2 Version 2.4.2.0 software and the ICDD PDF-2 Release 2015 RDB database, with a detection limit of 1%. To further confirm the phase analysis results obtained from the XRD, selected area electron diffraction (SAED) was also utilized. This analysis was conducted using a FEI Talos F200X transmission electron microscope (TEM) with an X-FEG source and maximum accelerating voltage of 200 kV. The TEM test was performed in a cross-sectional view, with an electron-transparent lamellas prepared by a focused ion beam (FIB) in a FEI Scios 2 Dual Beam system.

3. RESULTS AND DISCUSSION

The Cu-Fe-P alloy was studied to evaluate how different thermomechanical processing modes affect its mechanical and structural properties within the Cu-Fe-P ternary system. Fe and P were used as the alloying elements in concentrations of 0.003 wt% and 0.014 wt%, respectively. The results of the chemical composition analysis of the synthesized alloy are shown in Table 1. Elements such as S, Si, Se, Ag, Pb, Mn and Cr were detected in traces.

Table 1. *Chemical composition of key elements in the Cu-Fe-P alloy*

Element	Cu	P	Fe
wt%	99.98	0.0139	0.0028

The XRD pattern of the Cu-Fe-P alloy with a strain rate of 75% is shown in Figure 1. The spectrum shows that the Cu-Fe-P alloy has the Cu diffraction peaks corresponding to the pure Cu and copper oxide Cu_2O. The diffraction lines for Cu are represented by the peaks at 2θ angles of 42.91°, 51.84°, and 74.96°, corresponding to Cu (111), (200), and (220) crystal planes, respectively. Considering the low concentrations of Fe and P (0.003 wt% and 0.014 wt%, respectively), no diffraction peaks corresponding to Fe, P, or their possible compounds were detected, as the instrument detection limit is approximately 1%. In contrast to a diffractogram, where all peaks correspond to pure Cu in the Cu-Fe-P alloy, Zhang et al. [16] studied a series of Cu-20Fe-xP alloys. In their study, the Fe concentration was kept constant at 20 wt%, while the P concentration varied from 0.05 to 1.0 wt%. Their diffractogram revealed peaks corresponding to the Fe3P compound with reflections at (321) and (411), as well as the α-Fe phase with reflections at (110) and (200). Additionally, the low-intensity peaks in the spectrum corresponding to Cu_2O indicate the onset of oxidation, influenced by the material properties and its exposure to the atmospheric oxygen.

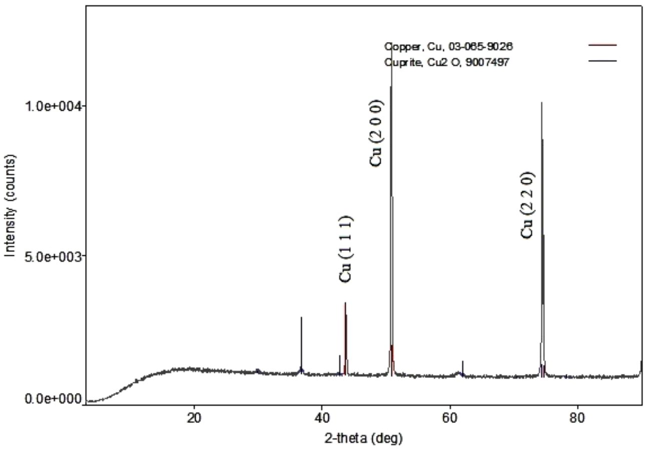

Figure 1. *XRD analysis of the Cu-Fe-P alloy with a deformation degree of 75%*

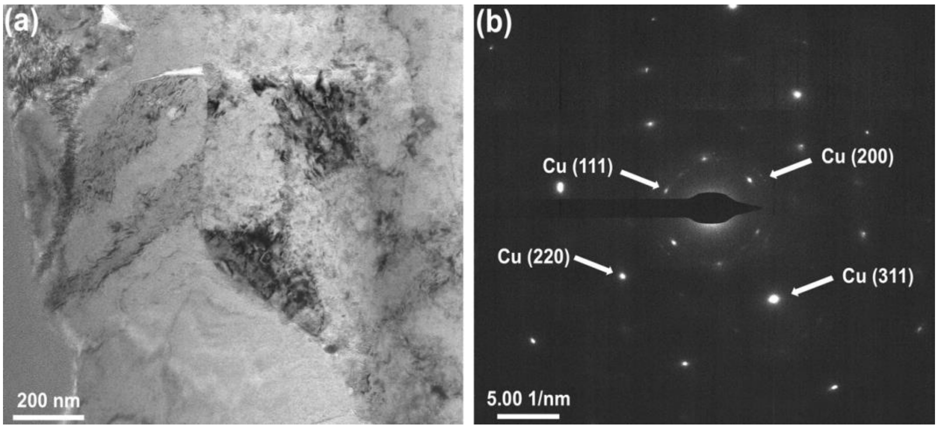

Figure 2. *TEM analysis of the Cu-Fe-P alloy with 75% deformation: (a) bright-field TEM micrograph and (b) SAED diffraction pattern*

Figure 2 presents a comprehensive overview of the microstructure and phase composition of the Cu-Fe-P alloy with 75% deformation, as elucidated through the TEM analysis. This is illustrated through a representative bright-field micrograph and the selected area electron diffraction image. The SAED pattern reveals the bright spots, indicating a highly crystalline structure. The observed reflections correspond to the cubic Cu phase, consistent with the XRD results. The hkl values, obtained from both XRD and TEM analyses, align with those listed in the reference pattern card PDF# 01-089-2838 (radiation wavelength 1.540590 Å), as detailed in Table 2.

Table 2. *Values of a plane hkl listed in the reference pattern card PDF# 01-089-2838*

d(Å)	***i***	***h***	***k***	***l***
2.08715	999	1	1	1
1.80753	428	2	0	0
1.27811	171	2	2	0
1.08998	156	3	1	1

4. CONCLUSION

The Cu-Fe-P alloy, after being melted, homogenized, extruded, and cold-rolled, was analyzed using the diffraction techniques. Comparative results from the XRD and TEM analysis revealed that the observed reflections matched the cubic structure of the Cu phase. The desired mechanical and structural properties were achieved through the specific mechanical and thermal treatments: extrusion with 75% deformation, recrystallization annealing at 470°C for 150 minutes, and subsequent cooling in a water-alcohol mixture. Despite using different methods, the diffraction results were consistent, confirming the alloy properties.

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