Influence of Alloying Elements on Cyclic Oxidation Behavior of (Cu/Al) Alloys

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Abstract. This study is conducted to extend the effective life of Cu/Al alloy. The used samples were (Cu/9.5%Al),(Cu/9.5%Al/5%Ni),(Cu/9.5%Al/5%Ni/0.9%Mn),(Cu/9.5%Al/5%Ni/4.5%Fe), (Cu/9.5%Al/5%Ni/0.9%Mn/4.5%Fe),(Cu/9.5%Al/5%Ni/0.9%Mn/4.5%Fe/0.1%Y),(Cu/9.5%Al/5%Ni/0.9%Mn/4.5%Fe/0.2%Ge) by using powder metallurgy these samples were prepared, the cyclic oxidation test was conducted in the presence of air in a programmable furnace (typeVBF-1200X-H8-USA) at 250℃ and 800℃ for 50 hours at 5-hours cycle. It was represented by the weight-gain of all examined samples that the oxidation rate increased as the temperature increased. It appears that cracking or spalling of oxides was at high temperatures such it found the weight gain of the base alloy (Cu/9.5%Al) was (12.09*10⁻³ g/cm²) at 250℃ otherwise, it was (5.98*10⁻² g/cm²) at 800℃ for the same alloy. This is attributable to the expected behavior of oxides when stressed. The addition of Ni, Mn, & Fe to (Cu/9.5%Al) caused a relative increase in resistance to cyclic oxidation, while the addition 0.1% Y to (Cu/9.5%Al/5%Ni/0.9%Mn/4.5%Fe) caused the lowest oxidation rate as compared with other alloys, it showed that the reduction in weight gain (165.13%) relative to (Cu/9.5%Al) the highest weight gain at 250℃, and it was (285.22%) relative to (Cu/9.5%Al/5%Ni) which has highest weight gain at 800℃. Both alloying elements Y and Ge represented a major change in cyclic oxidation resistance.

Keywords. Cu/Al Alloys, Cyclic Oxidation.

1. Introduction

Nowadays, the various copper alloys find their application in almost any field, from telecommunication and automotive to distribution and power generation, offshore and marine industries, as well as applications of shape memory [1]. Particularly in offshore and marine technology, Cu/Al and Cu/Ni alloys were used as tubing, propeller, and fitting material [2]. Copper-aluminum (Cu/Al) alloys are referred to as aluminum bronzes, so they constitute an essential category of engineering materials recognized for good strength and corrosion resistance. In order to ensure optimal properties, the properties are primarily based on microstructure, so an understanding of the phase transformations that occur is necessary. The binary diagram is complicated, but the most important reaction for the commercial importance of binary alloys is the eutectoid that takes place at 565 °C. Alloys that contain Fe and Ni are also of significant commercial significance. Each of these elements already significantly influences the relationships between the phases and affects the alloy properties. Single-phase α alloys are consisting of less than 8% aluminum. The
formation of \( \alpha \) dendrites starts with solidification. The freezing range seems to be low, but as a single phase, segregation is also not pronounced as well as the alloy solidifies. An alloy that contains 10% aluminum solidifies when it solidifies \( \beta \). \( \alpha \) continues to deposit from the \( \beta \) when the temperature decreases to around 930 °C showed in Figure1: The copper-aluminum (Cu/Al) binary phase diagram. The amount increases as the temperature decreases. Directly comparable to that found in a (60/40) brass, a Widmanstätten structure is formed. The remaining \( \beta \) continues to decompose to either a lamellar eutectoid, \( \alpha + \gamma_2 \) at 565 °C. The reaction is regulated by diffusion and only moves to completion when the alloy is kept at temperatures far less than that of the eutectoid for a substantial amount of time, even during slow cooling. To enable this reaction to occur, typical casting rates will be too rapid, so \( \beta \) is retained. By a process without needing diffusion, retained \( \beta \) transforms into such a hard acicular martensitic form. The martensitic transition is not included in the equilibrium diagram since it is a non-equilibrium phase. However, it does have important implications also for the diagram's mechanical properties. However, it has significant consequences for the mechanical properties of the alloy. Concerning corrosion resistance and mechanical properties, the eutectoid \( \alpha + \gamma_2 \) structure has some unfavorable characteristics and is thus avoided much further than possible like most commercial applications [3].

![Figure 1. The Cu-Al binary phase diagram [3].](image)

In recent years, a multitude of mechanistic information has become obtainable. This data covers a wide variety of phenomena, such as evaporation of oxide or metallic species, mass transport through oxide scales, the function of mechanical stress in oxidation, the growth of scales with more than one oxidant in complex environments, and the essential relationships between alloy microstructure, composition, and oxidation [4]. In 2003 Jingbo Yan et al. [5] was examined the oxidation of a Cu-rich and a Ni-rich single-phase ternary alloy containing about 5 % Al at 800°C and 900°C under 1 atm O₂. The Ni-rich alloy's behavior is similar to that of a binary Ni/ aluminum alloy with a similar Al content at both temperatures. The development of an external NiO layer combined with internal oxidation of Al₂O₃. The (Cu-rich) ternary alloy shows a high tendency to form protective Al₂O₃ scales, even though its behavior is borderline between non-protective & protective. In fact, at 800°C, after an initial rapid reaction stage during which all the alloy components are oxidized, this alloy capable of making a continuous layer of Al₂O₃ at the base of the scale, which prevents the internal oxidation of Al. By comparison, at 900 °C, the innermost Al₂O₃ layer Repeated rupturing accompanied by healing follows, so that internal oxidation of Al is only partly eliminated. As a result, the copper-rich ternary alloy's corrosion kinetics at 900°C is much faster than at 800°C and very similar to those of pure Cu & of Al-dilute binary Cu/Al alloys. Probable purposes for the la tendency of the Curich alloy to form external Al₂O₃ scales than the Ni-rich alloy had
been examined. In 2006 Y. Niu et al. [6] had been investigated at 800°C in 1 atm O₂ the oxidation of the three Cu–xCr–4Al and the three Cu–xCr–2Al alloys (x~0.4,8 at.%). Oxidation of a binary Cu/Al alloy consists of 2.2 at.% Al produced external scales collected mainly of copper oxides with small rates of Al-rich oxide in the inner region, while the internal oxidation of aluminum was almost absent. The addition of 3.9 % Cr to this alloy was able to decrease the oxidation rate but was scanty to prevent Cu's oxidation. Reciprocally, the addition of 8.1 % Cr to the same binary alloy promoted the relatively rapid formation of a protective alumina layer in contact with the metal substrate, with a simultaneous broad decrease in the oxidation rate, producing a form of third-element effect. On the contrary, all the Cu–xCr–4Al alloys formed an internal alumina layer after an initial stage during which all the alloy components were oxidized, so that the only effect of the presence of chromium was to decrease the duration of the rapid initial stage. The 3rd-element effect due to chromium additions to Cu–2Al is related to a transition from the formation of external scales composed of mixtures of Cu & Al oxides to the external growth of alumina rich scales as an effect of a thermodynamic destabilization of Cu- oxides associated with the formation of solid solutions between Cr₂O₃ & Al₂O₃. In 2005 Gabriel Plascencia et al. [7] studied the elevated-temperature oxidation resistance of Cu & Cu/Al alloys in the air was investigated using thermogravimetric analysis. Upon heating in air, Cu starts to oxidize at temperatures above 400°C noticeably. It was found that as the temperature increased, more Al was required in order to protect the Cu. Alloying with 4 wt.% Al leads to a remarkable improvement in oxidation resistance. The atmosphere used to heat the samples to the required test temperatures had a noticeable impact on the subsequent oxidation rates. When heated in argon before being oxidized, Cu alloys with 3 wt. % & 4 wt. Al showed good oxidation resistance with rates thousand times less than that of pure Cu at 1,000°C. However, when these alloys were heated in air, they have been much less successful in supplying oxidation resistance. In 2014 W.A. Badawya et al. [8] studied the effect of adding Aluminum, Zinc, and Nickle to Cu either as a single alloying element in binary alloys such as bronzes &brasses, it was researched the effect of the presence of two additional elements with Copper so that to enhance the electrochemical behavior of these alloys in solutions of chloride content, (Cu%10.0Al%10.0Ni), (Cu%10.0Al%10.0Zn) and (Cu%10.0Ni%10.0Zn) had been used so the surfaces of these alloys examined by EDAX-SEM analysis. The surface analysis showed the participation of the various additional elements in the passive film according to the alloy's content and that the Cl⁻ is deeply permeated in the alloy. This research is characterized by preparing a (Cu/9.5Al) alloy by using powder technology (P/M). The addition of alloying elements such as germanium and yttrium to test the resistance to cyclic oxidation in air at two different temperatures(250&800) and compare with the base alloy.

2. Experimental

2.1. Samples preparation
The samples were inspected using the powders is shown in Table 1, where either particle size, purity, and source of the powders are also shown as follows:

| Powder | Average particle size (μm) | Purity % | Source |
|--------|----------------------------|----------|--------|
| Cu     | 5.03                       | 99.97    | Sky,Sipring Nanomaterials,Inc/USA |
| Al     | 20.07                      | 99.913   | Sky,Sipring Nanomaterials,Inc/USA |
| Ni     | 4.736                      | 99.87    | Sky,Sipring Nanomaterials,Inc/USA |
| Fe     | 17.74                      | 99.926   | Sky,Sipring Nanomaterials,Inc/USA |
| Mn     | 29.49                      | 99.882   | Sky,Sipring Nanomaterials,Inc/USA |
| Ge     | 3.782                      | 99.938   | BDH Chemicals Ltd/England         |
| Y      | 4.489                      | 99.94    | BDH Chemicals Ltd/England         |
To achieve the objectives of this study, several mixtures were prepared from different powders. The main mixture includes (Cu /9.5% Al) had been prepared then Ni, Mn, Fe, Y, and Ge were added to (Cu /9.5% Al) mixture with different weight percentages (0.9%,4.5%,0.1%, and 0.2%) to explain the influence of these elements on the cyclic oxidation resistance of the prepared alloys. Table 2. shown the prepared alloys used in this study.

| Alloy No. | Composition (weight parent of powders) |
|----------|----------------------------------------|
| A        | Cu + 9.5% Al                           |
| B        | Cu + 9.5% Al + 5% Ni                   |
| C        | Cu + 9.5% Al + 5% Ni+0.9%Mn            |
| D        | Cu + 9.5% Al + 5% Ni+4.5% Fe           |
| E        | Cu + 9.5% Al + 5% Ni+0.9%Mn+4.5% Fe    |
| F        | Cu + 9.5% Al + 5% Ni+0.9%Mn+4.5% Fe+0.1%Y |
| G        | Cu + 9.5% Al + 5% Ni+0.9%Mn+4.5% Fe+0.2%Ge |

Using a small amount of acetone (10 ml for each 30g of the mixture), the blending of elementary powders has been done using the electric roll mixer. The maximum rotating speed was (90 rpm), while the total mixing time was about six hours. The hydraulic press for uniaxial pressing had used to compact green samples for physical and microstructural tests. The cold uniaxial pressing cylindrical die had used to prepare samples with dimensions (20 mm in diameter and 8 mm in height) for microstructural examination. In order to obtain the optimal compression pressure, it was used to 750 MPa as compression pressure. After the compaction, the green compacts of all samples had placed in the furnace shown in Figure 2 under the inert gas environment with a continuous stream of argon. The pressure of the gas inside the furnace had controlled by a graduated gage from 1 to 25 bar as shown in Figure 3. This have been done to isolate the samples from oxygen and thus prevent oxidation at elevated temperatures during the sintering process, with the thermal cycle shown in Figure 4. which include green heating compact from room temperature up to 550°C with heating rate 10°C/min., 120 min is the time of staying. With the same heating rate, increase the furnace's temperature up to 950°C with 180 min as staying time, then the samples have been left in the furnace to slow cooling.

![Figure 2. Vacuum Chamber Furnace-VBF-1200X-H8-USA.](attachment:figure2.jpg)

![Figure 3. Gages from 1 to 25 bars.](attachment:figure3.jpg)
Several heat treatments have been used in this study, including solution heat treatment (Betatizing) thermal cycle, as shown in Figure 5; then, the thermal cycle of aging heat treatment (post quench), as shown in Figure 6.

2.2. Chemical composition test
For conducting chemical analysis of samples, they were taken to the Ministry of Science and Technology for conducting the tests using analytical instruments /Spectro-XEPOS. The chemical composition of the samples with impurities shown in Table 3. Samples A, B, and G are the Cu/Al alloy without and with alloying elements addition, respectively. The samples were sectioned into.
### Table 3. Chemical composition for all samples with impurities concentration.

| Alloy Code | Cu%  | Al%  | Ni%  | Fe%  | Mn%  | Ge%  | Y%  | Mg%  | Mo%  | Ti%  | W%  | Co%  | Sr%  |
|------------|------|------|------|------|------|------|-----|------|------|------|-----|------|------|
| A Bal.     | 9.1308 | 0.0888 | 0.0609 | 0.0406 | ---  | ---  | 0.035 | 0.05  | 0.0111 | ---  | 0.0318 | 0.015 |
| B Bal.     | 9.1039 | 6.075 | 0.0668 | 0.0379 | ---  | ---  | 0.035 | 0.055 | 0.0140 | 0.015 | 0.0331 | 0.018 |
| C Bal.     | 9.1121 | 5.927 | 0.0756 | 1.259  | ---  | ---  | 0.034 | 0.043 | 0.0120 | 0.015 | 0.0347 | 0.02  |
| D Bal.     | 9.0693 | 6.079 | 4.378 | 0.0699 | ---  | ---  | 0.035 | 0.041 | 0.0118 | 0.014 | 0.0439 | 0.018 |
| E Bal.     | 9.1356 | 5.762 | 4.367 | 1.311  | ---  | ---  | 0.037 | 0.045 | 0.0155 | 0.015 | 0.0295 | ---  |
| F Bal.     | 9.1791 | 5.613 | 4.422 | 1.118  | 0.095 | 0.036 | 0.075 | 0.0135 | ---  | 0.0184 | 0.05  |
| G Bal.     | 9.2546 | 6.009 | 4.864 | 1.999  | 0.179 | 0.035 | 0.0621 | 0.0110 | ---  | 0.0176 | 0.013 |

2.3. Scanning electron microscopy (SEM)

Scanning electron microscope observation is also used to detect the microstructure of etched samples. The images captured with magnifications 200X, 210X, 214X, 182X, 208X, 180X and 20 KV. The prepared samples has been wet grinding using (320, 400, 600, 800, 1000, 1200, 1500, 2000, 2500, 3000, 5000). the papers of grit silicon carbide using grinding wheel machine so then polishing by using polishing paper and diamond with 1-3 μm as particle size, then cleaned these samples with water and dried by hot air. The Samples are then etched with etching solution (2.5 g FeCl₃ .6H₂O + 48 ml CH₃OH + 10 ml HCl) ferric chloride acid / methanol /hydrochloric acid [9] for 10 sec. the samples after that are ready to use for scanning electron microscope observation. SEM examination had been done in the University of Technology /Production Engineering and Metallurgy.

2.4. Cyclic oxidation test

The cyclic oxidation test is a measure of the adhesion and plasticity of the developing oxide layer on the different samples. This test was performed for all oxides formed on the alloy surfaces used. Cyclic oxidation tests were carried out in a programmable furnace (typeVBF-1200X-H8-USA) at temperatures 250°C, 800°C where the sample weight is first recorded and then quickly removed from the oven to cool to room temperature in air and then returned to the oven to see how cracking or spalling occurs in the oxide layer, Test conducted on all alloys samples for 50 hours. Each heating cycle includes heating in the furnace for five hrs. At the test temperature and cooling in still air. In Figure 7. Shown samples after cyclic oxidation test. Phases analysis has been done for all samples after cyclic oxidation test at 250°C, 800°C, X-ray diffraction test carried out at Iran/Mashhad/Tavous Research institutePOW3040/60with XPertSoftware type Philips /Holland.

![Figure 7. Samples used after cycle oxidation test.](image-url)
whereas C, S, O, and Si are considered as impurities in alloys composition. On the other hand, low amounts of Y in sample F and Ge in sample G would be absent in these samples’ elemental compositions, which may be due to the limitations of EDS as an analytical tool. However, a clear improvement in resistance to oxidation resistance at high temperatures for this sample is evidence of these two elements.

Figure 8. A1. SEM image, A2. EDS analysis for alloy A after the aging process

Figure 9. B1. SEM image, B2. EDS analysis for alloy B after the aging process
Figure 10. C₁. SEM image, C₂. EDS analysis for alloy C after the aging process

Figure 11. D₁. SEM image, D₂. EDS analysis for alloy D after the aging process
Figure 12. E1. SEM image, E2. EDS analysis for alloy E after the aging process

Figure 13. F1. SEM image, F2. EDS analysis for alloy F after the aging process
Cyclic oxidation tests in the air at 250 °C and 800 °C it was carried out to study the cyclic oxidation resistance of alloys A, B, C … G. The oxidized behavior of alloys, in general, was parabolic. These tests are a measure of the determination of the types of developing oxides, growth rate, adhesion, and plasticity, as shown in Figures 15 and 16 below:

**Figure 14.** G1. SEM image, G2. EDS analysis for alloy G after the aging process.

**Figure 15.** Parabolic fitted results of weight gain vs. time plot for all samples (A, B … G) cyclic oxidized in air at 250°C for 50 hours at 5-hours cycle.

**Figure 16.** Parabolic fitted results of weight gain vs. time plot for all samples (A, B ..., G) cyclic oxidized in air at 800°C for 50 hours at 5-hours cycle.
From Figure 15, it can be noted that samples are sequenced as A…E, G, F since weight gain is reduced by the increase in the addition of alloying elements to alloy A. Sample A (Cu/9.5%Al) has the highest weight gain; this is expected due to aluminum which can protect itself by creating a stable layer from aluminum oxide (Al₂O₃) as found in XRD pattern of cyclic oxidation test at 250°C. However, it is brittle, and it is not very adhesive due to which cracking and fracture occur in the oxide layer when the sample is exposed to cyclic oxidation. Then, it begins to build a protective layer again. Therefore, over time, the process's intensity will increase when the temperature rises to 800 °C, as shown in Figure 16. As the cyclic oxidation effect of 800°C can be more severe than 250°C, it can be explained that thick oxide layers are more susceptible to breakage due to the many defects. The value of high thermal stress placed on the oxide layer is in good agreement with those given by [10]. Sample B suffers from a continuous increase in oxidation rate over time with an increase in oxidation temperature, but the oxidation rate is lower than that in sample A and at 250°C, but at 800°C, it is higher than sample A due to the presence of alumina on the surface of this sample. The weight gain of samples C, D, E is clearly decreasing due to alloying elements Mn and Fe, and this indicates the importance of adding some elements to improve the general characteristics of the alloy, including cyclic oxidation resistance. We note that weight gain and the protective layer building for these samples are much lower than the samples of A and B, which contain elements that impact forming a protective layer with the best possible adhesion. The samples F and G have proved to be more efficient than other samples. Sample F showed cracks when the temperature increased, but it was less than the other samples due to Yttrium's effect in building the protective oxide layer and improving the adhesives of it; this result is in agreement with those in [11]. The sample G has improvement in the oxides layer; this improvement is due to germanium the oxide is formed GeO₂ as found in the XRD pattern of cyclic oxidation test at 800°C. This oxide has surface protection that is much better than an alumina especially because it improves surface elasticity. It is not breakable; this result agrees with those in [12], [13]. Table 4 shows weight gain and reduction in weight gain % for all samples relative to sample A (highest weight gain alloy at 250°C) and relative to sample B(highest weight gain at 800°C). Although there is a difference in the resistance of cyclic oxidation of samples used, they all have good oxidation resistance due to the formation of protective layers, which are the XRD pattern results shown in the figures below. The oxides growing on the surfaces of all the samples used in the research could rebuild themselves after testing the cyclic oxidation. In Figure 17, the XRD pattern from the surface of samples A...G after cyclic oxidation test in the air at 250°C, it is found α-Cu, Al₂O₃ peaks while some intermetallic compound AlCu₅, AlNi present on A, B,..., G whereas Fe₂Al peak indicated on samples E and F, in sample D exist CuMn₂O₄ spinel compound. Also, Y₃Al₅O₁₂, Fe₂O₃ present in sample F, in sample G, the formation of CuMn, FeO, and NiFe₂O₄ was confirmed through X-ray diffraction analysis in sample G. Whereas in Figure 18 from the surface of alloys A...G after cyclic oxidation test in the air at 250°C and 800°C, while in the XRD pattern of samples A, B...G representing α-Cu, Al₂O₃, AlNi, MnAl peaks. Therefore, Fe₂O₃ exists in the XRD pattern for samples D, F, G, while it has found GeO₂ in G.

| Alloy | Weight gain (g/cm²) At 250 °C | Reduction in weight gain % At 250 °C | Weight gain (g/cm²) At 800 °C | Reduction in weight gain % At 800 °C |
|-------|-------------------------------|--------------------------------------|-------------------------------|--------------------------------------|
| A     | 12.09*10⁻³                   | 5.98*10⁻²                            | ----                          | 87.46                               |
| B     | 11.5*10⁻³                    | 11.21*10⁻²                           | 5.13                          | ----                                 |
| C     | 9.23*10⁻³                    | 9.21*10⁻²                            | 30.99                         | 21.72                               |
| D     | 8.45*10⁻³                    | 8.22*10⁻²                            | 43.08                         | 36.37                               |
| E     | 7.34*10⁻³                    | 4.32*10⁻²                            | 64.71                         | 159.49                              |
| F     | 2.91*10⁻³                    | 1.76*10⁻²                            | 315.46                        | 563.93                              |
| G     | 4.56*10⁻³                    | 2.91*10⁻²                            | 165.13                        | 285.22                              |
Figure 17. XRD pattern from the surface of samples A, B...G after cyclic oxidation test in the air at 250°C (A) alloy A, (B) alloy B, (C) alloy C, (D) alloy D, (E) alloy E, (F) alloy F, and (G) alloy G.
Figure 18. XRD pattern from the surface of alloys A, B … G after cyclic oxidation test at 800°C. (A) alloy A, (B) alloy B, (C) alloy C, (D) alloy D, (E) alloy E, (F) alloy F, and (G) alloy G.
4. Conclusions
   1. All samples showed adequate resistance to cyclic oxidation at 250°C and 800°C for 50 hours, then the oxidation increases with increasing temperature. So, it was found that the lowest weight gain at 250°C was 2.91*10^-3 (g/cm²) for sample F as compared to sample A, which showed higher weight gain equal to 12.09*10^-3 (g/cm²), while the lowest weight gain, 1.76 *10^-2 (g/cm²), was for sample F at 800°C as compared to sample C, which showed higher weight gain equal to 11.21*10^-2 (g/cm²).
   2. The samples showed the formation of cuprous oxide Cu₂O, Al₂O₃, and Fe₂O₃, NiO, Cu Mn₂O₄, and the formation of other phases when exposed to cyclic oxidation at temperatures of 250°C and 800°C for 50 hours.
   3. The oxides that grew on all the samples used in the research could rebuild themselves after testing for cyclic oxidation.
   4. Thin oxide layers showed higher resistance to cyclic oxidation than thicker layers.
   5. The addition of yttrium and germanium to samples F and G, respectively, represented a significant shift in cyclic oxidation resistance compared to all other samples used.

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