Effect of Alloying Elements on the Erosion–Corrosion Behaviour of Cu-Based Alloys

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Abstract. This study used a powder metallurgy (P/M) to prepare a sample of a Cu / 9.5 % Al alloy and add alloying elements (9.5 % Al five % Ni 0.9 % Mn 4.5 % Fe 0.2 % Ge) to it. The Cu / five % Ni alloy has been used for comparison with other research alloys. The powders were mixed for six hours and pressed under 750 MPa. The samples were sintered at 550 °C for two hours, and then the temperature was raised to 950 °C for a period of 180 min with a heating rate of ten °C / min under an inert gas environment with a continuous stream of argon. It was then left to cool inside the furnace. After this, several heat treatments were used. Some tests were carried out, such the scanning electron microscopy (SEM), energy dispersion spectrometer (EDS), X-ray diffraction (XRD), Vickers micro-hardness and the erosion–corrosion test. The alloy Cu / five % Ni showed the least hardness. The addition of Ni, Mn and Fe to the Cu / 9.5 % Al alloy increased the hardness, and these additions caused a relative increase in the resistance to erosion–corrosion. However, it also caused a large increase in the resistance to erosion–corrosion when adding Ge to the alloy Cu / 9.5 % Al / five % Ni 0.9 % Mn 4.5 % Fe 0.2 % Ge when immersed in seawater. Additionally, it demonstrates a high hardness. These improvements were attributed to an enhancement of the properties of alloy-based Cu – 9.5 % Al, which is used in the marine industry.

1. Introduction
Copper, as well as alloys, is a metal that is recognisable by the public. It is easily recognised by its colour, and it has been used since the Bronze Age for tools, arms, jewels and housewares at the reference point of metallurgy. These were commonly used for their thermal properties. Nowadays, it is still the main metal for electrical connections, as well as for wires in devices, appliances and microelectronics. Regarding the mechanical properties, copper is also an fcc-type metal [1]. Aluminium bronzes (Cu / Al) are best known for their high strength and excellent resistance to corrosion. Their resistance to stress-corrosion fatigue exceeds that of austenitic stainless steels. They can be easily welded and can be machined or ground, although good lubrication and cooling are necessary for a fine surface finishing. Common uses involve marine hardware, shafts, pumps and valve components for seawater handling and sour mine water, as it has no oxidising acids or fluids from the manufacturing process. The strong wear resistance of the alloys makes it the perfect choice for heavy-duty sleeve bearings and machine tools [2]. Its properties are strongly dependent on the microstructure, as an understanding of the phase transformations that occur is important to ensure optimum properties. The binary diagram is complicated, but the most important reaction for
commercially applicable binary alloys is the eutectoid at 565 °C. Alloys that contain Fe and Ni are also of great commercial significance. Each of these elements have an important effect on the process relationships and affect the alloy properties [3]. Alloys containing less than eight % aluminium are single-phase α alloys. Solidification starts when α dendrites form. The range of freezing is short, but there is no evident segregation, and the alloy solidifies that as a single phase. As β solidifies, it creates an alloy of ten % Al. Therefore, when the temperature falls by approximately 930 °C, the β, as shown in Figure 1, starts to deposit. This is shown by the binary phase diagram of Cu-Al [2]. When the temperature falls, the amount increases. A Widmanstätten structure is created, which is directly comparable to that found in brass of 60 / 40. The remaining β starts to decompose at 565 °C into a lamellar eutectoid, α + γ2. The reaction is regulated by diffusion, and only stops when the alloy is kept at temperatures far below that of the eutectoid for a significant amount of time, as may occur during slow cooling. To allow that reaction to occur, normal casting rates are too rapid. Therefore, β is retained. The retained β transforms the martensite into a hard, acicular form by a method that does not require diffusion. As it is a phase of non-equilibrium, the martensitic changes are not included in the equilibrium diagram. However, it does have important implications for the mechanical properties of the diagram and the alloy. With respect to the mechanical properties and corrosion resistance, the eutectoid α+ γ2 structure has some unacceptable characteristics. Therefore, it is often avoided as it is not practicable for most industrial applications [4].

Figure 1. The copper-aluminium (Cu-Al) binary phase diagram [2].

In 2005, Al-Haidary J. T. [5] studied the erosion–corrosion behaviours of α-brass with some additives (0.2 % Al, 0.2 % Te and two % Ge) while subjected to a jet of tap water + 3.5 % NaCl. The results suggested the roles of these elements in improving the erosion–corrosion resistance. In 2009, Faisel et al. [6] prepared alloys (Cu – 9.5 % Al) that were used in marine propellers using the powder metallurgy...
technique. They added alloying elements to the base alloy (five % Ni / one % Mn / four % Fe / three % Si) and carried out several tests, such as hardness and corrosion / erosion tests. The results showed that the alloying elements increase the corrosion / erosion when added to Cu 9.5 % Al. The improvement ratio was 488.9 % when the heat treatment was done, and the hardness value was 185 HV. In 2014, W.A. Badawya et al. [7] studied adding Al, Zn and Ni to copper as a single alloying element in binary alloys, such as bronzes and brasses. This was to research the effect of mixing two alloying elements with Cu, as well as to investigate the electro-chemical behaviour of these alloys in solutions consisting of chloride. Cu- / 10.0 Al / 10.0 Ni, Cu / 10.0 Al / 10.0 Zn and Cu / 10.0 Ni / 10.0 Zn were used, and these alloys' surfaces were examined using the EDAX and SEM analyses. The surface analysis showed the participation of the various alloying elements in the passive film according to the alloy constituents. It also showed that the chloride ions were deeply permeated in the alloy. In 2019, Vu Anh Tuan et al. [8] studied the effect of Ni on the mechanical and microstructure of the Cu alloy. This study shows the results of nickel components on the mechanical and microstructure properties of the Al / Cu alloys. With the heat treatment process, it was heated to 850 °C, quenched in water, and then tempered at 350 °C for two hours. As the sample had added Ni, it had phases of nickel with aluminium and iron. These phases made the mechanical properties of the sample better than the sample without nickel. In the samples that had nickel added, the hardness was higher than those without Ni, as it was 10.92 HRB. The results of abrasion showed that the Ni alloyed samples presented better than those without Ni. This study aims to produce an alloy of Cu / 9.5 Al using powder technology and other alloys by adding alloying elements to study the impact of seawater for use in marine industries.

2. Experimental Programme

2.1. Preparation of the Samples’ Stages

The samples were prepared using the powders shown in Table 1, where the particle size of the powders, the purity and source are shown as follows:

| Powder       | Average particle size (μm) | Purity % | Source                                           |
|--------------|---------------------------|----------|--------------------------------------------------|
| Copper       | 5.03                      | 99.97    | Sky, Sipring Nanomaterials, Inc / USA            |
| Aluminium    | 20.07                     | 99.913   | Sky, Sipring Nanomaterials, Inc / USA            |
| Nickel       | 4.736                     | 99.87    | Sky, Sipring Nanomaterials, Inc / USA            |
| Iron         | 17.74                     | 99.926   | Sky, Sipring Nanomaterials, Inc / USA            |
| Manganese    | 29.49                     | 99.882   | Sky, Sipring Nanomaterials, Inc / USA            |
| Germanium    | 3.782                     | 99.938   | BDH Chemicals Ltd / England                     |

2.1.1. Preparation of the Blended Mixtures

Using a small amount of acetone (ten ml per 30g of the mixture) to avoid agglomeration of the mineral powders and oxidation problems, the elementary powders were blended using the electric roll mixer. The maximum rotating speed was 90 rpm, while the total mixing time was approximately six hours. The mixtures included Cu + five % Ni and Cu + 9.5 % Al, and Ni, Mn, Fe and Ge were added to the Cu + 9.5 % Al mixture with different weight percentages (0.9 %, 4.5 % and 0.2 %) to explain the influence of alloying elements on the hardness and erosion–corrosion resistance of the prepared alloys. The prepared alloys that were used in this study are shown in Table 2.
Table 2. Mixtures and their compositions, as prepared by this study.

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

2.1.2. Compaction of Powders
The hydraulic press for uniaxial pressing was used to compact the green samples. The cold uniaxial pressing cylindrical die was used to prepare samples with dimensions of 20 mm diameter and eight mm height for use in the erosion-corrosion test. The steel die interior walls were graphite lubricated. Exactly 750 MPa compression pressure was used to determine the optimum compression pressure to give a lower green porosity and higher green density.

2.1.3. Sintering
After the compaction procedure, the green compacts of all samples were placed in a furnace (type VBF-1200X-H8-USA) under an inert gas environment with a continuous stream of argon. The pressure of the gas inside the furnace was controlled by a graduated gage from one bar to 25 bar. This was done to isolate the samples from oxygen to prevent oxidation at elevated temperatures during the sintering process. This used the thermal cycle shown in Figure 2 and included the following steps:

1- From room temperature, the heating green compacts up to 550 °C with a heating rate of ten °C / min.
2- The duration is 120 min.
3- The temperature of the furnace is increased to 950 °C with the same heating rate.
4- The duration is 180 min.
5- The samples are left to slow cool in the furnace.

The sintering of all samples was successful with low porosity. No distortions occurred in the samples’ shapes, and no fusion in any elements occurred. This is due to the high purity of the powders that were used, as well as the accurate thermal cycle [9].

Figure 2. Thermal cycle used in sintering.
2.1.4 Heat Treatment After Sintering
Several heat treatments have been used in this study, including solution heat treatment (betatising) and aging heat treatment (post-quench). The details of this treatment are provided in the following paragraphs:

2.1.4.1. Solution Heat Treatment (Betatising)
After sintering, the samples are solution treated by heating the samples up to 850 °C for 60 min. Next, the samples (B, C, D) are rapidly quenched into ice water at three °C–six °C to avoid decomposition from the β phase into the equilibrium phases, including α + γ2, which is the eutectoid reaction at 565 °C for aluminium bronze in the alloy phase diagram. The solution heat treatment thermal cycle is shown in Figure 3.

2.1.4.2. Aging Heat Treatment
Aging heat treatment was done after the quenching heat treatment to age several intermetallic compounds, such as γ2, in the microstructure to modify their properties. The aging heat treatment temperature was 200 °C for 30 hrs for the prepared samples (B, C, D), followed by cooling in iced water so as to not begin the decomposition phase during slow cooling [9]. Increasing the ageing time leads to an increase in the γ2 precipitates size and a loss of coherency [10]. The thermal cycle is shown in Figure 4.

![Figure 3. Solution heat treatment (betatising) thermal cycle.](image)

![Figure 4. Thermal cycle of aging heat treatment.](image)
2.2. Sample Tests

2.2.1. Chemical Compositions

The chemical composition examination is shown in Table 3 below. It analyses the chemical compositions and concentrations of impurities for all samples.

| Alloy Code | Cu % | Al % | Ni % | Fe % | Mn % | Ge % | Mg % | Mo % | Ti % | W % | Co % | Si % |
|------------|------|------|------|------|------|------|------|------|------|-----|------|------|
| A          | Bal. | 0.0755 | 4.554 | 0.0537 | 0.0434 | --- | 0.034 | 0.025 | 0.0130 | 0.015 | 0.0313 | 0.016 |
| B          | Bal. | 9.1308 | 0.0888 | 0.0609 | 0.0406 | --- | 0.035 | 0.05 | 0.0111 | --- | 0.0318 | 0.015 |
| C          | Bal. | 9.1356 | 5.762 | 4.367 | 1.311 | --- | 0.037 | 0.045 | 0.0155 | 0.015 | 0.0295 | --- |
| D          | Bal. | 9.2546 | 6.009 | 4.864 | 1.999 | 0.179 | 0.035 | 0.0621 | 0.0110 | --- | 0.0176 | 0.013 |

2.2.2. Scanning Electron Microscopy SEM and EDS analyses

The SEM observation was used to detect the microstructure of the etched samples (sample A after sintering and samples B, C and D after aging process). The images were captured with magnifications 200X, 208X, 180X, and 20 KV. Wet grinding was done on the samples that were prepared using 320, 400, 600, 800, 1000, 1200, 1500, 2000, 2500, 3000, and 5000 grit silicon carbide papers with a grinding wheel machine. They were then polished using a diamond with one to three μm as the particle size and polishing paper. After that, they were cleaned with water and dried using hot air with an etching solution of 2.5 g FeCl3·6H2O + 48 ml CH3OH + ten ml HCl ferric chloride acid / methanol / hydrochloric acid [11] for approximately ten sec etching for the samples. Then, the samples were ready for the scanning electron microscope observation. The SEM examination was done in the University of Technology Production Engineering and Metallurgy.

2.2.3. X-Ray Diffraction Analysis (XRD)

The phase’s analysis of the prepared samples was based on the X-ray diffraction technique and microstructures examination using an optical microscope with different magnifications. The test was done after the aging treatment was carried out on all samples (A, B–H) to see the effect of heat treatment. The X-ray diffraction tests were carried out at the Faculty of Materials Engineering and in the General Company for Engineering Inspection and Qualification in Baghdad in Iraq. The Mini flex2 device was held at angles of (2θ) = ten–90 deg. with a step (0.02 deg.) This gave more precise details. In addition, the test was done for all samples after the erosion–corrosion test. The X-ray diffraction tests were carried out at the Iran / Mashhad / Tavous Research institute POW3040/60 with XpertSoftware type Philips / Holland.
2.2.4. Micro-Hardness Test
The Vickers micro-hardness test was done for all samples with a load of 500 g and holding time of 20 sec [9], along with X200 as the magnification for the light optical microscope that was attached using the Vickers instrument. Four readings were taken for each sample because there could have been pores that were located on the sample’s surface that would affect the hardness. The measurements were done automatically and taken directly from the digital screen of the device.

2.2.5. Erosion–Corrosion Test
Erosion is known as a mechanical wear process, i.e. the loss of materials from solid surfaces by the impacting or impingement of liquid, solids or gases. The erosion–corrosion testing device was manufactured and designed during this work according to (G 73) ASTM [12], as shown in Figure 5. The erosion–corrosion device consists of the chamber and comprises a cylindrical plastic tank. The dimensions of the tank are 50 cm in height and 30 cm in diameter. The various media is directly supplied at high pressures, and the vertical position of the sample’s face through the jet nozzle (d = 0.75 mm) uses a pump to suck the different media from the plastic tank chamber with \( \frac{3}{4} \) horse power. From Teflon, the single-phase electric motor (chemical pump) was made, so the distance between the sample and nozzle was ten mm. The chemical pump is resistant to the chemical solution and slurry. The valves and pipe joints that are connected to the chamber with the chemical pump are made from PVC plastic.

![Figure 5. The erosion–corrosion device.](image)

3. Results and Discussion
An SEM observation was done for alloy A after sintering at 550 °C for 120 minutes and 950 °C for 180 minutes. It was also done for alloys B, C and D after the aging heat treatment, where the samples were heated up to 200 °C for approximately 30 hours, and then rapidly quenched in iced water. According to Figures 6, 7, 8 and 9, sample A contains the \( \alpha \) phase, while in samples B, C and D, the phases are identified as the \( \alpha \) phase and contain the intermetallic compound \( \gamma_2 \). This conclusion was also corroborated by an
XRD analysis. The elemental compositions of these samples were obtained using SEM–EDS. According to Figure 4, the elemental compositions in sample A are Cu and Ni. Figures 5, 6 and 7 show the alloy composition in three samples that are made of the bronze alloy. The main alloying elements of all samples are Cu and Al. Nickel is detected as the main concentration in samples C and D. Manganese is considered to be an alloying addition in samples C and D, while iron is present in the elemental compositions for samples C and D as an alloying addition. However, C and O are considered impurities in the alloy composition. On the other hand, low amounts of Ge in sample D are absent in the elemental compositions of this sample, which may be due to the limitations of EDS as an analytical tool. However, a clear improvement in the resistance to erosion–corrosion for this sample is evidence of germanium.

Figure 6. A1. SEM image, A2. EDS analysis for sample A after the sintering process.

Figure 7. B1. SEM image, B2. EDS analysis for sample B after the ageing process.
Figure 8. C₁. SEM image, C₂. EDS analysis for sample C after the ageing process.

Figure 9. D₁. SEM image, D₂. EDS analysis for sample D after the ageing process.
The XRD pattern of alloy A, as shown in Figure 10, shows that the existing phases were \( \alpha \)-Cu, Ni and \( \text{Cu}_{3.8}\text{Ni} \) after the sintering process at 550 °C for 120 minutes and 950 °C for 180 minutes. Figures 11 to 13 show the XRD pattern for alloys B, C and D after the aging heat treatment in which the samples were heated up to 200 °C for approximately 30 hours, and then rapidly quenched in iced water. It was found that the aging heat treatment led to precipitation in the intermetallic compound \( \gamma_2 \text{AlCu}_3 \) and \( \alpha \) phase. Regarding the other phases that can be formed between copper, aluminium, nickel, iron and germanium, they can have a significant influence on the alloys’ properties. However, they did not appear in the X-ray diffraction pattern, as which suggests that they may be outside the limits of the XRD detection.
Figure 12. XRD pattern for sample C after aging.

Figure 13. XRD pattern for sample D after aging.
Table 4 provides the hardness values of each individual sample. Sample A has the lowest hardness value compared to B, C and D. This is expected because the relationship between copper and nickel is complete solubility, and at any percentage, they completely dissolve each other. As is known at five % nickel, the tensile strength is less likely to be present for this alloy, which increases after this value. Therefore, we see that this alloy has lower hardness [15]. Adding the alloying elements Ni, Mn and Fe will increase the strength because heat treatment increases hardness. The addition of Ge has the same effect as the previous results, as the addition of germanium will be deposited freely, thereby helping to refine the grains. Therefore, it will increase the hardness compared to alloys A, B and C. At the same time, it will improve the plasticity of the alloy [16].

| Sample | Vickers Micro-hardness g/μm² | Improvement % |
|--------|-----------------------------|--------------|
| A      | 65                          | ....         |
| B      | 139                         | 113.85       |
| C      | 189                         | 190.77       |
| D      | 200                         | 207.69       |

The effect of the chemical composition and the duration of exposure to the sea water jet on the weight loss of the samples A–D are shown in Figure 14. Figure 15 shows the erosion–corrosion rate test in solution 3.5 % NaCl of the samples A–D for ten hours. For samples A, B and C, we note in Table 5 that the weight loss of sample B (0.088943 g.cm⁻²) is the lowest compared with A (0.063663 g.cm⁻²) and C (0.080945 g.cm⁻²) in the steady state. This was expected because sample B contains 9.5 % Al; therefore, there is a rich oxide layer of Al₂O₃. The rate of weight loss for samples A and B changed with the duration of exposure to the seawater jet, where the incubation period was not observed. When the test began, the surface of the samples hardness by drops of water. Following that, spalling occurs, which causes an acceleration in the weight loss. This reached a great value in the first quarter hour of the test. It was followed by a deceleration period, which represents a decrease in the rate of weight loss because the sample surface is rough in this period with grooves. These are then filled with salty water, and the jet of salty water will contact the water rather than the alloy. This was followed by a steady state, which has a stable weight loss. On the other hand, sample C was characterised by the appearance of the incubator period and a lower rate of weight loss. It reached the steady-state period faster than samples A and B. This sample showed a good resistance to erosion–corrosion, as there was no weight loss during the first half of its exposure to seawater due to the combined effect of iron with manganese. It also showed a clear increase in erosion–corrosion resistance, and this result is in agreement with Callcut [15]. Alloy D showed excellent resistance to both the erosion–corrosion and the weight loss compared with the others. The longest incubation period resulted in the lowest rate of weight loss and the least time to achieve the steady-state period compared to the other samples. This is due to the presence of a germanium element that can improve mechanical properties, give high ductility to the oxide layer [16] and improve corrosion resistance, which is in good agreement with Rosalbino [17].
Figure 14. The effect of chemical composition and the exposure period of the seawater jet on the weight loss of samples A–D for ten hours.

Figure 15. The erosion–corrosion rate test in the solution 3.5 % NaCl of the samples A–D for 10 hours.
Table 5. Weight loss for all samples and their improvement.

| Sample | Weight loss g .cm\(^{-2}\) | Improvement % |
|--------|-----------------------------|---------------|
| A      | 0.089                       | -----         |
| B      | 0.064                       | 39.71         |
| C      | 0.032                       | 177.97        |
| D      | 0.022                       | 308.84        |

4. Conclusion

The XRD analysis and SEM observation showed that the formed phase in the sintered sample (Cu-five % Ni) was (α), whereas in the aged samples of Cu- 9.5 % Al with the alloying addition of elements Ni, Mn, Fe and Ge, the formed phases were α + γ\(_2\). The alloy D showed an increase in hardness by 207 % as compared to the alloy A which showed the lowest hardness. Alloy D showed a decrease in weight loss by 308.84 % compared to alloy A, which showed the highest weight loss after being subjected to an erosion–corrosion test in laboratory-prepared seawater for ten hours. The incubation period (latency) for the sample D was long, whereas for the rest of the research alloys, it was either absent or short. The addition of germanium to alloy D represented a significant shift in the erosion–corrosion resistance compared to the other alloys.

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