

# Corrosion investigation of rapidly solidified Cu- Al-Ni alloy in NaCl solution

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## Corrosion investigation of rapidly solidified Cu-Al-Ni alloy in NaCl solution

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### Abstract

*The corrosion behavior of the Cu-Al-Ni alloy ribbons obtained by rapid solidification was investigated in 0.9% NaCl solution (pH 7.4) at temperatures of 24, 37 and 50 °C. Measurements have been performed by electrochemical methods such as monitoring the open circuit potential, linear and potentiodynamic polarization methods. The influence of chloride ions concentration on corrosion of Cu-Al-Ni ribbons has also been investigated in 0.1% and 1.5% NaCl solution. Results of the investigations have shown that increase in chloride concentration and electrolyte temperature leads to increase the corrosion current density and decrease the polarization resistance values which mean higher corrosion attack on Cu-Al-Ni alloy.*

*Microscopic images have shown significant pitting corrosion damages on the Cu-Al-Ni alloy surface. Increasing the electrolyte temperature increases the surface damages of the electrode due to more intense corrosion. SEM surface images of Cu-Al-Ni electrodes have confirmed that with elevation of chloride concentration a more intense corrosion attack occurs. EDS surface analysis indicated dominant percentage of copper, chlorine and oxygen on the surface, indicating the formation of copper oxide and chloride as the major corrosion products on the surface. The presence of a small percentage of aluminum indicates its distribution in the form of aluminum oxide and chloride in the surface layer.*

**Keywords:** Cu-Al-Ni alloy, melt spinning method, corrosion, electrochemical methods

### Introduction

Copper alloys are extremely used materials due its favorable mechanical properties and excellent electrical and heat conductivity [1-2]. A special group of copper alloys are shape memory alloys which has characteristic features such as superelasticity and shape memory effect owing to the reversible martensitic transformation [3-6]. These alloys can return to their previously defined shape or size when they are subjected to appropriate treatment. This behaviour is related with the solid state transformation of martensite to austenite and vice versa [2-4]. Because of its features, shape memory alloys find their application in transport industry, medicine, robotics etc. [5,7-9]. Due to its superb mechanical properties and biocompatibility, the most commercially used shape memory alloys is NiTi alloys [2,10]. Cu-Al-Ni alloys have some considerable advantages over Ni-Ti alloy such as less difficult melting, casting and composition control, better machinability, better work/cost ratio and better stability of the two way shape memory effect [2]. However, Cu-Al-Ni and Cu-Zn-Al shape memory alloy are brittle susceptible to intergranular fracture due to its coarse grain structures and high elastic anisotropy [4,11,12].

There are few ways how to influence to these negative properties, such as thermomechanical procession, use of micro-alloy elements for grain refinement and use rapid solidification process in

alloys production [12-15]. Generally there are four advantages of rapid solidification over the slow conventional solidification techniques. These are an ability to form metastable phases, increasing the solubility above the equilibrium solubility, decreasing the segregation of additions and refining the microstructure [13-16].

In this investigation the corrosion properties of Cu-Al-Ni alloy ribbons, produced by rapid solidification process (melt spinning), has been investigated in deaerated NaCl solution at different temperatures of electrolyte and different chloride concentrations.

## Experimental

Rapidly solidified ribbons of Cu-Al-Ni with composition of 83 wt.% Cu, 13 wt.% Al and 4 wt.% Ni were manufactured with the single roll melt spinning apparatus. The cast precursors were inserted into the graphite crucible and inductively melted in Ar atmosphere and sprayed through the nozzle into the cooled rotating copper wheel. The Cu-Al-Ni alloy ribbon samples for the electrochemical measurements were prepared by cutting to the appropriate dimensions and then soldered on an insulated copper wire to gain good electrical contact. Soldered joint points are insulated with Polirepar S protective mass to prevent the evaluation of galvanic corrosion in contact with the electrolyte. Because of the small thickness of the Cu-Al-Ni ribbons, mechanical treatment by grinding and polishing could not be performed, but the surface of the electrode was processed by ultrasonic degassing in ethanol, after which it was washed with deionized water and immersed in the electrolyte. Electrochemical investigations were performed on Cu-Al-Ni ribbon samples in a 0.9% NaCl solution pH = 7.4, on a Princeton Applied Research potentiostat/galvanostat (Model 273A).

The influence of electrolytic temperature on corrosion behavior of Cu-Al-Ni alloy ribbons was investigated in deaerated 0.9% NaCl solution at 24 °C, 37 °C and 50 °C while the influence of chloride concentration were investigated in 0.1 %, 0.9 % i 1.5 % NaCl solution, pH = 7.4, T = 37 °C. Prior and during the immersion period solution was deaerated by bubbling with high purity argon and held at a constant temperature. A saturated calomel electrode (SCE) was used as a reference and the Pt sheet as a counter electrode. The open circuit potential measurements (EOC) were made immediately after electrode immersion in a time period of 60 min whereupon linear polarization measurements were performed in a potential region of  $\pm 20$  mV around EOC, with the scan rate of 0.2 mV s<sup>-2</sup>. The final electrochemical method used was potentiodynamic polarization method in a potential region of -250 mV vs. EOC to 700 mV.

After the potentiometric polarization measurements the surface of the electrode were washed in deionized water ultrasonically, dried in the desiccator and then visualized by Canon IXUS1000 camera in macro mode and the light microscope with magnification of 100 times. A more detailed surface analysis was performed with Scanning Electron Microscope Tescan Vega 5136 MM paired with Energy Dispersive Spectroscopy (SEM/EDS).

## Results and discussion

The time dependence of open circuit potential values for Cu-Al-Ni alloy in 0.9% NaCl at electrolytic temperatures of 24 °C, 37 °C and 50 °C is shown in Figure 1, while Figure 2 shows the time dependence of open circuit potential values for Cu-Al-Ni alloy in 0.1%, 0.9% and 1.5% NaCl solution (pH = 7.4 and T = 37 °C). Investigations were performed by recording the potentials every 30 seconds over a 60 minute time period.



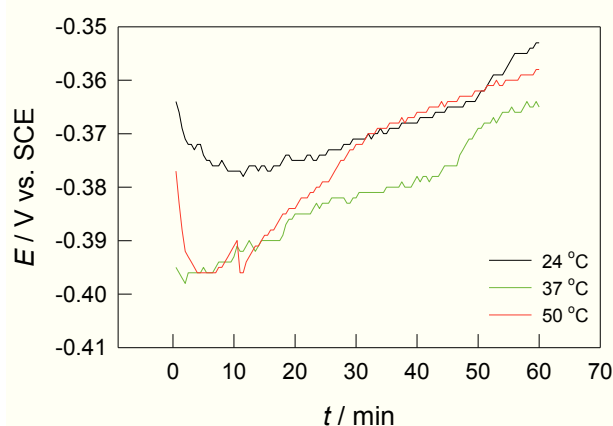


Figure 1. Variation of the open circuit potential with time for the Cu-Al-Ni alloy in 0.9% NaCl solution at 24 °C (-), 37 °C (-)

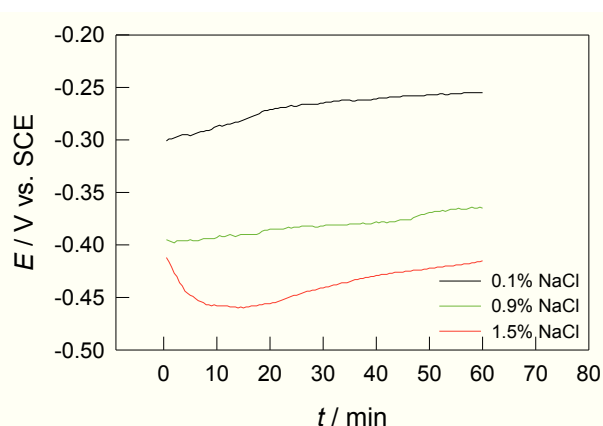


Figure 2. Variation of the open circuit potential with time for the Cu-Al-Ni alloy in NaCl solution 0.1% (-), 0.9% (-) and 1.5%

From the Figure 1 it is apparent that in the initial time period immediately after the electrode immersion in the electrolyte there is a negation of the open circuit potential values, followed by its positivation. This trend was observed for all electrolyte temperature investigated. After 60 minutes, no significant differences in Cu-Al-Ni alloy open circuit current values were observed at different temperature of the solution.

From the time dependence of the open circuit potential for Cu-Al-Ni alloys in 0.1%, 0.9% and 1.5% NaCl solution (Figure 2.) it is clearly visible that the electrolyte concentration significantly influences the values of the open circuit potential of the Cu-Al-Ni alloy and that with increasing concentration of chloride ions potential values becomes increasingly negative.

Immediately upon the completion of the measurement of open circuit potential, linear polarization measurements were made in the narrow area of potentials around the open circuit potential, and the results are shown in Figures 3 and 4.

With the increase of the electrolytic temperature, the slope of the linear curves of the curve decreases, indicating a reduction in the polarization resistance values. As the polarization resistance represents the corrosion resistance of the material, lowering the values of polarization resistance means that with increasing temperature the corrosion of the alloy becomes more intense. Similar observations were also noticed in the investigation of electrolyte concentration (Figure 4) where it was found that increasing the electrolyte concentration leads to a decrease in the linear curve parts. The picture also shows a significant difference in the value of the potentials area where the measurements were made, as a result of the negative potential of the open circuit with elevated electrolyte concentrations. From the inclination of the linear curve parts, the values of polarization resistance are determined and are shown in Table 1 along with the data from potentiodynamic polarization measurements.



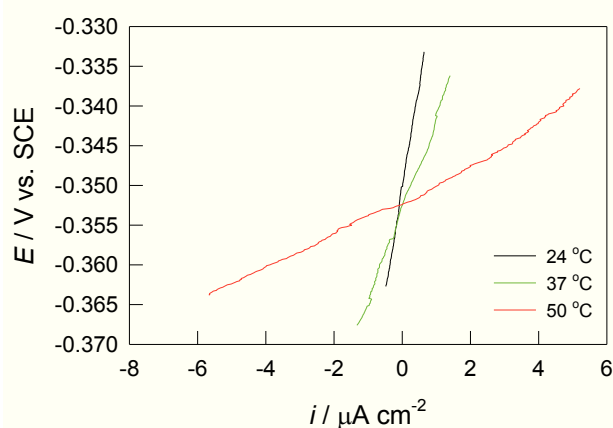


Figure 3. Results of the investigation by linear polarization method for Cu-Al-Ni alloy in 0.9% NaCl solution at 25 °C (-), 37 °C (-)

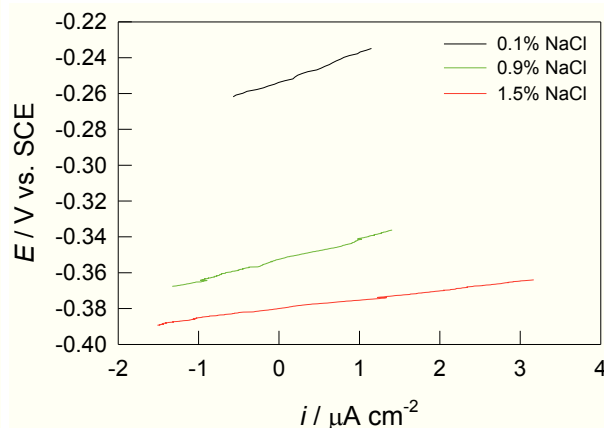


Figure 4. Results of the investigation by linear polarization method for Cu-Al-Ni alloy in NaCl solution 0.1% (-), 0.9% (-) and 1.5% (-)

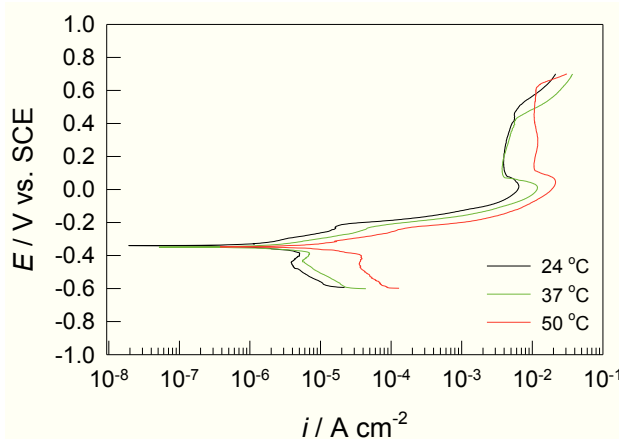


Figure 5. Potentiodynamic polarization curves for Cu-Al-Ni alloy in 0.9% NaCl solution at 25 °C

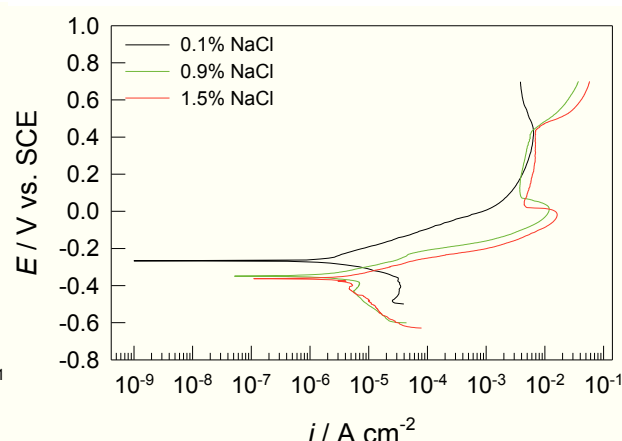


Figure 6. Potentiodynamic polarization curves for Cu-Al-Ni alloy in NaCl solution 0.1% (-), 0.9% (-) and 1.5% (-).

Figure 5 shows potentiodynamic polarization curves for Cu-Al-Ni alloy in 0.9% NaCl solution at electrolytic temperatures of 24 oC, 37 oC and 50 oC. The shape of the polarization curves is almost the same at all examined electrolyte temperatures, which indicates the same behavior of the alloy. Increasing the temperature leads to an increase in cathodic and anodic current density while the corrosion potential does not change, indicating more intense corrosion at higher electrolyte temperatures.

Figure 6 shows the results of the the potentiodynamic polarization investigation for the Cu-Al-Ni alloy in 0.1%, 0.9% and 1.5% NaCl at  $T = 37$  oC. From the Figure can be seen that the behavior of the Cu-Al-Ni alloy in the 0.1% NaCl solution is different from the behavior in the 0.9% and 1.5% NaCl solutions. The differences is visible in the anodic branches of the polarization curves, because for the measurements in higher concentration of NaCl solution anodic branches can be divided onto three distinct areas: Tafel area, pseudo-passive area and area where current density increases [2]. According to literature, [2,16,17], in the linear Tafel's region, copper dissolving and forming of soluble  $\text{CuCl}_2$  complexes occur; in the active-passive area there is a decrease in current density due to the formation of hard-soluble corrosion products on the surface made up of copper oxide and chloride, [2,17,18] and also traces of aluminum oxide/ hydroxide [2], while the third area is characterized by a re-growth of anodic current density indicating that the resulting surface compounds do not form a compact protective film and the alloy dissolution is continued by the formation of

soluble Cu (II) compounds [18]. On the anodic part of the polarization curve for Cu-Al-Ni alloy in a 0.1% NaCl solution, the pseudopassive area appears at much more positive potential (about 0.5 V) where there is already a rise in the anodic current density on curves obtained at higher chloride concentrations. This difference is due to the low concentration of chloride ions, which is why the corrosion processes are considerably smaller. The corrosion parameters from the polarization curves are given in Table 1.

Table 1. Corrosion parameters obtained from potentiodynamic polarization measurements and linear polarization measurements

Conditions	$i_{\text{corr}} / \mu\text{A cm}^{-2}$	$E_{\text{corr}} / \text{V}$	$R_p / \text{k}\Omega \text{ cm}^2$
24oC; 0.9% NaCl	1.67	-0.266	26.379
37oC; 0.9% NaCl	2.0	-0.347	11.484
50oC; 0.9% NaCl	12.64	-0.347	2.306
37oC; 0.1% NaCl	2.3	-0.265	15.684
37oC; 1.5% NaCl	1.6	-0.352	5.178

From the Table 1 it can be seen that increase in electrolyte temperature as well as increase in chloride ion concentration increases the values of corrosion currents density and decrease values of polarization resistance which indicate more intense corrosion of the CuAlNi alloy.

After potentiometric polarization measurements, Cu-Al-Ni electrode surface was investigated using a Canon IXUS 1000 digital camera in macro mode and an optical microscope to determine the surface condition. The results are shown in Figures 7-11.



Figure 7. Cu-Al-Ni electrode after polarization measurements in 0.9% NaCl solution,  $T = 24 \text{ oC}$ ,  $\text{pH} = 7.4$ : a) macro image and b) light microscope image with magnification of 50 times.

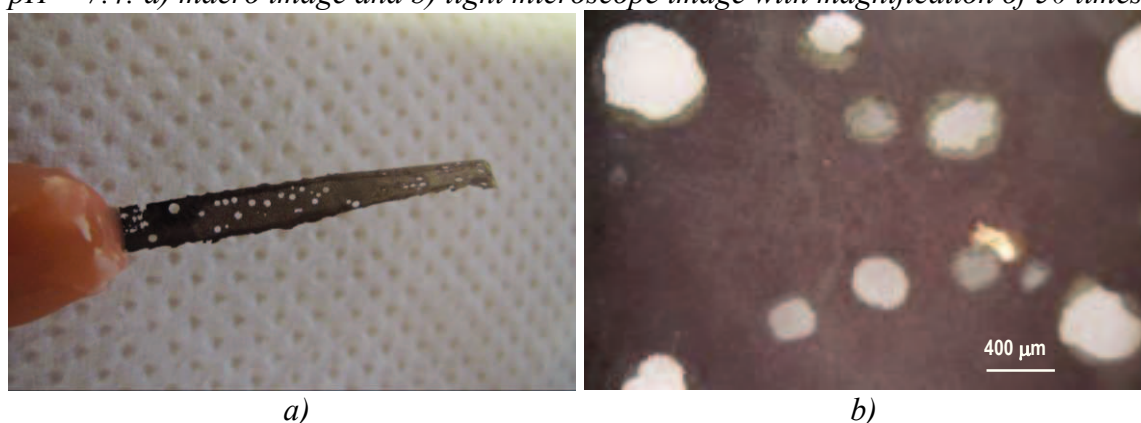


Figure 8. Cu-Al-Ni electrode after polarization measurements in 0.9% NaCl solution,  $T = 37 \text{ oC}$ ,  $\text{pH} = 7.4$ : a) macro image and b) light microscope image with magnification of 50 times.

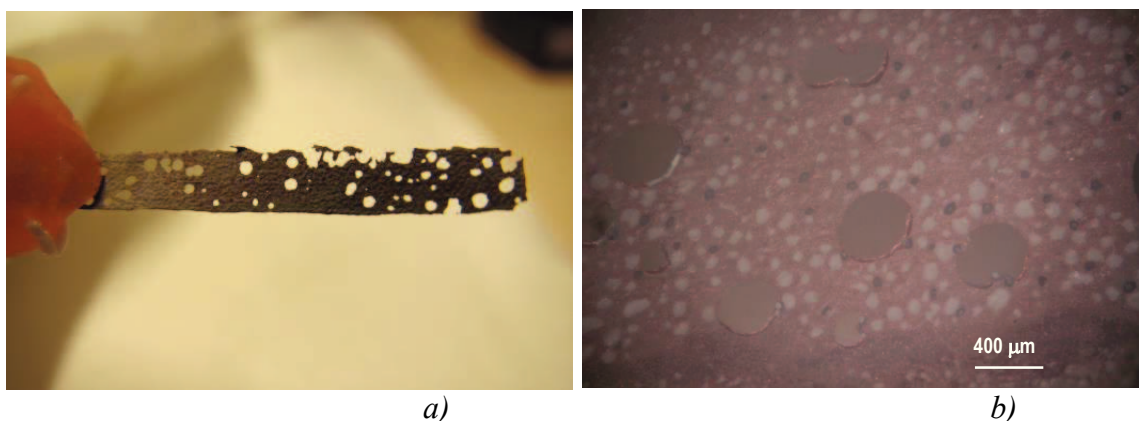


Figure 9. Cu-Al-Ni electrode after polarization measurements in 0.9% NaCl solution,  $T = 50$  oC,  $pH = 7.4$ : a) macro image and b) light microscope image with magnification of 50 times.

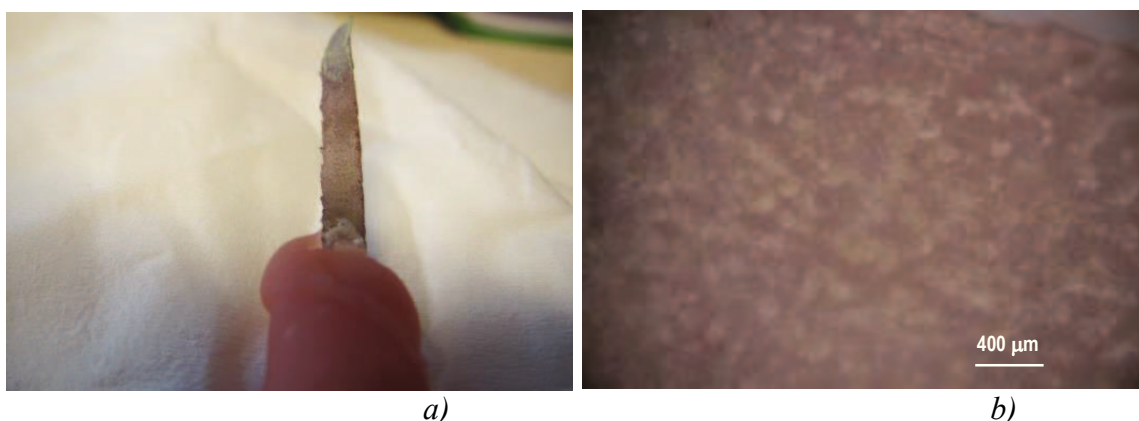


Figure 10. Cu-Al-Ni electrode after polarization measurements in 0.1% NaCl solution,  $T = 37$  oC,  $pH = 7.4$ : a) macro image and b) light microscope image with magnification of 50 times.

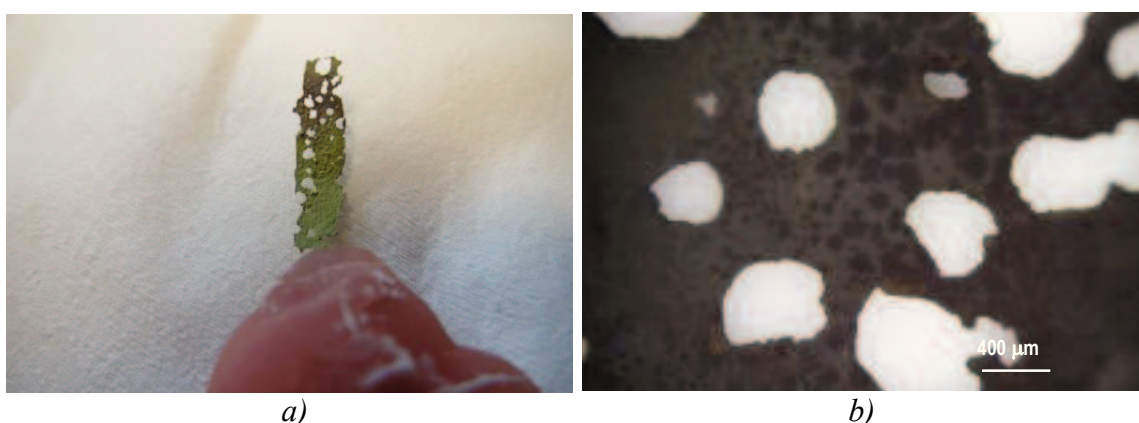
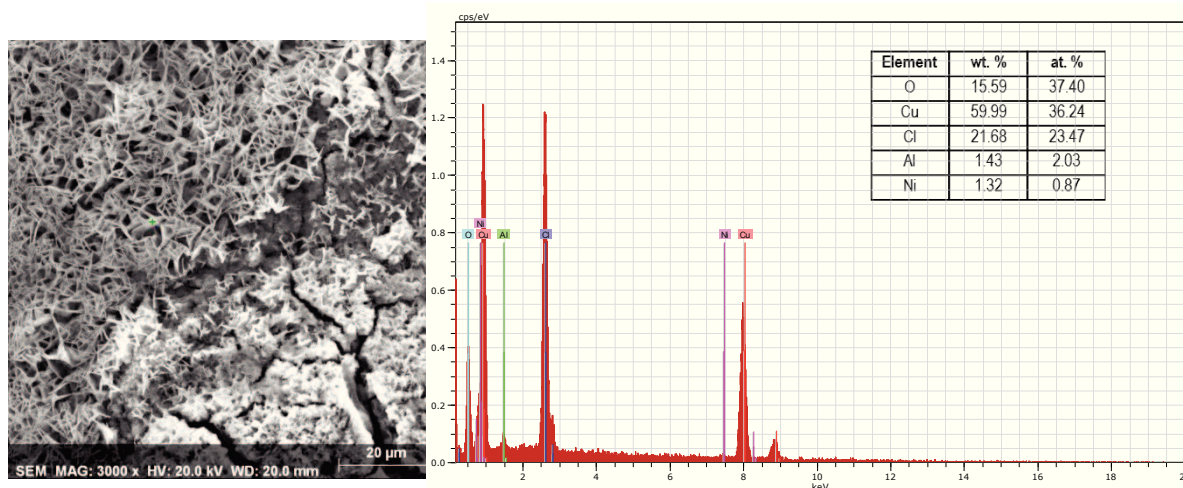


Figure 11. Cu-Al-Ni electrode after polarization measurements in 1.5% NaCl solution,  $T = 37$  oC,  $pH = 7.4$ : a) macro image and b) light microscope image with magnification of 50 times.

Intense pitting corrosion was observed on all Cu-Al-Ni electrodes except on the electrode exposed to the 0.1% NaCl solution. It is apparent that corrosive destruction of the electrode increases with increasing electrolyte temperature as well as with increasing its concentration.

More detailed information on the electrodes surface condition after electrochemical measurement was obtained by scanning electron microscope, while the elemental composition was determined by EDS method, and the results are shown in Figures 12-13.

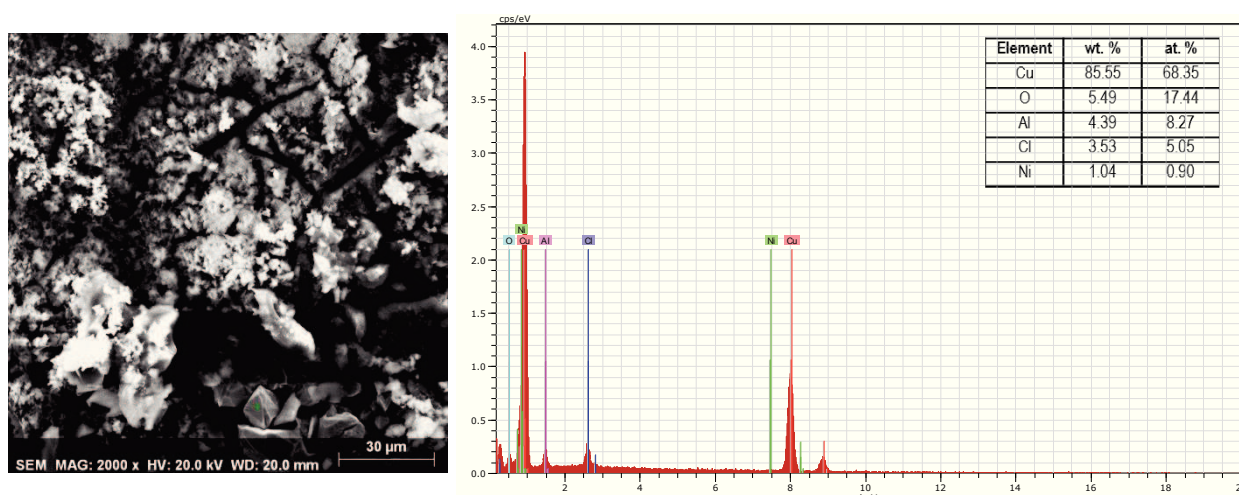




a)

b)

Figure 12. a) SEM images of the Cu-Al-Ni surface after potentiodynamic polarization measurement in 0.1% NaCl solution; b) EDS analysis



a)

b)

Figure 13. a) SEM images of the Cu-Al-Ni surface after potentiodynamic polarization measurement in 1.5% NaCl solution; b) EDS analysis

Significant surface damages can be seen on SEM images caused by corrosion in the chloride electrolyte, which are much more pronounced on electrode exposed to higher concentration of chloride ions. EDS surface analysis showed the presence of Cu, Cl and O as the dominant surface elements, from which it can be concluded that the surface corrosion products consist mainly of copper oxide and chloride, but also contained a small amount of aluminium oxide, as Al is also detected by EDS analysis.

## Conclusions

Open circuit potential for the Cu-Al-Ni alloy shifts in the negative direction by increasing the chloride concentration in the solution, while the increase in the electrolyte temperature does not significantly change the potential of the open circuit.

Increase in electrolyte temperature as well as concentration of chloride ions leads to decrease the polarization resistance values and increase the values of corrosion currents density, which indicates a more intense corrosion process.

Macro images of the electrode surfaces after polarization measurements with a digital camera and an light microscope with a magnification of 50 times show that in 0.1% NaCl no pitting corrosion occurs on Cu-Al-Ni alloy while at higher concentrations of NaCl there is an appearance of pitting corrosion on the electrode surface. Increasing the electrolyte temperature increases the surface damages of the electrode due to more intense corrosion.

SEM surface images of Cu-Al-Ni electrodes have confirmed that with elevation of chloride concentration a more intense corrosion attack occurs. EDS surface analysis indicated dominant percentage of copper, chlorine and oxygen on the surface, indicating the formation of copper oxide and chloride as the major corrosion products on the surface. The presence of a small percentage of aluminum indicates its distribution in the form of aluminum oxide and chloride in the surface layer.

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