

The corrosion study of Co-Cr-Ni alloy fabricated by powder metallurgy

Raudhah Othman, Shamsul Baharin Jamaludin, Mohd Nazree Derman

Abstract— Cobalt-based alloys have been widely used in biomedical application. In this research, various compositions of Co-Cr-Ni alloys were developed and produced by powder metallurgy route. This study was also aimed to investigate the corrosion behavior of Co-Cr-Ni alloys. The Co-Cr-Ni powders were first milled using planetary mill and compacted using uniaxial hand press to a pellet shape before being sintered using tube furnace. Immersion test was carried out in a water bath while maintaining the temperature of 37°C. Energy dispersive X-ray spectroscopy (EDS) was used for elemental analysis of the immersed samples. The result showed that sample with $\text{Co}_{91}\text{Cr}_4\text{Ni}_5$ composition has the lowest mpy result after 30 days immersed in 0.9% sodium chloride solution while sample with $\text{Co}_{57}\text{Cr}_{20}\text{Ni}_{23}$ composition recorded the highest mpy value.

Keywords: Co-Cr-Ni, immersion, weight loss, EDS

I. INTRODUCTION

Materials used in medicine should fulfill several strict conditions primarily imposed by biological restrictions: absence of harmful interactions with body cells, good compatibility with organ and, finally, suitable mechanical properties [1]. The success of a biomaterial in the body depends on factors such as the material properties, design, and biocompatibility of the material used, as well as factors not under control of the engineer, including the technique used by the surgeon, the health and condition of the patient, and the activities of the implant. Biocompatibility can be explained as an acceptance of an artificial implant by surrounding tissues and by the body as whole. The implant should be compatible with tissues in terms of mechanical, chemical, surface, and pharmacological properties [2-4].

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There are three categories of materials presently used in prosthetic devices which are metals, polymers, and ceramics. Biometals used in orthopedic implants include surgical grade stainless steel, cobalt-chromium alloys, titanium, and titanium alloys. Stainless steel is not suitable for a permanent implant because of its poor fatigue strength and liability to undergo plastic deformation, yet is still commonly used for nonpermanent implant such as internal fixation devices for fractures. Before the use of titanium, cobalt-based alloys had largely replaced stainless steel as materials for permanent implants. These alloys are generally more corrosion resistant owing to the formation of a durable chromium-oxide surface layer or passivation layer [5].

Chromium as well as molybdenum has an ability to improve the corrosion resistance of alloys. Besides they act as tungsten stabilize the hcp-structure of Co-matrix which is important to improve the mechanical properties, reduce the abrasive wear and lower the stacking faults energy. The formation of stacking faults diminishes the ductility of cobalt alloys. However, Co-Cr alloy experienced a low ductility which by alloy additions, their properties could be improved. By adding Ni, it can reduce the tendency to form stacking faults which improves ductility and stabilizes fcc-structure of Co-matrix [5].

An immersion test of a series of powder metallurgy Co-Cr alloy with nickel addition has been investigated in order to obtain the alloy with optimal corrosion properties. EDS in conjunction with immersion test is applied to study the corrosion behavior of Co-Cr-Ni alloys.

II. EXPERIMENTAL

Cobalt, chromium and nickel powders were mixed together in planetary ball mill (Recth, Pm100). The speed applied was 450rpm and rotated for 30 minutes. A series of ten different compositions of CoCr and CoCrNi were prepared (Table 1). This series was taken based on the study by Matkovic et al. (2004) through their paper entitled effects of Ni and Mo on the microstructure and some other properties of Co-Cr dental alloy.

Alloys of cobalt, chromium and nickel were fabricated using powder metallurgy technique and was compacted using a stainless steel die with 10 mm diameter using a uniaxial press. A pressure of 240 MPa at room temperature was applied to all samples. In powder pressing, the degree of compaction is maximized and fraction of void space is minimized by using coarse and fine particles mixed in appropriate proportions.

For uniaxial pressing as being applied, the powder is compacted in a metal die by pressure that is applied in a single direction. The formed piece takes on the configuration of die and platens through which the pressure is applied. A sintering operation is required after the pressing operation. During sintering the formed piece shrinks, and experiences a reduction of porosity and an improvement in mechanical integrity. These changes occur by the coalescence of the powder particles into a more dense mass in a sintering process [6].

The samples were sintered under argon atmosphere using tube furnace. The heating rate was set to 3°C / min until it reached 400°C and was soaked for 1 hour, which then the heating rate for sintering process was set up to 5°C / min until it reached 1000°C and was soaked for 2 hours.

An immersion test were applied to samples where the samples were first being ground using 240 grit SiC paper followed by 320, 400, 600, 800, 1000 and 1200 grit SiC paper and polished. The samples were then measured for their height, diameter and weight before placing them in a test tube filled with 0.9% sodium chloride solution and covered with aluminium foil. The prepared samples were then placed in the Digital Precise Shaking Water Bath (WSB-30) with the temperature maintained at 37°C for 30 days.

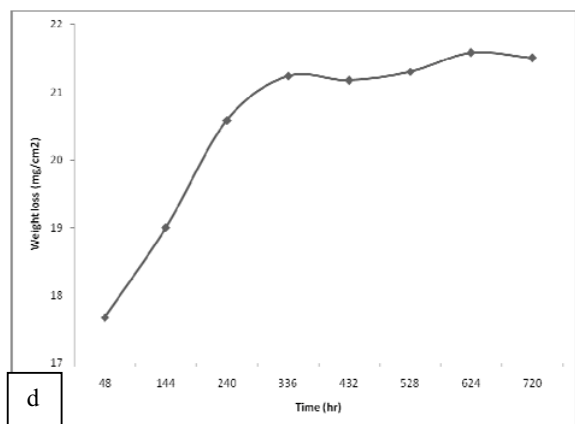
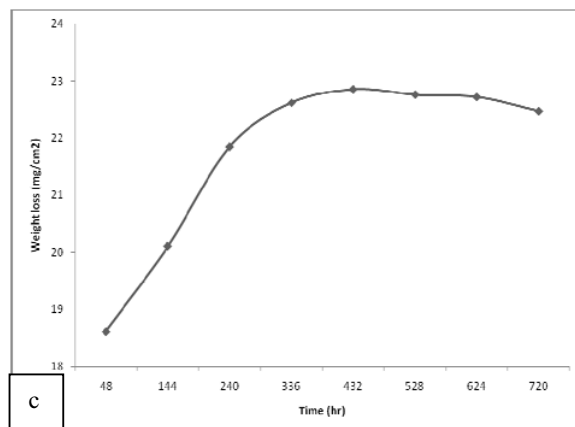
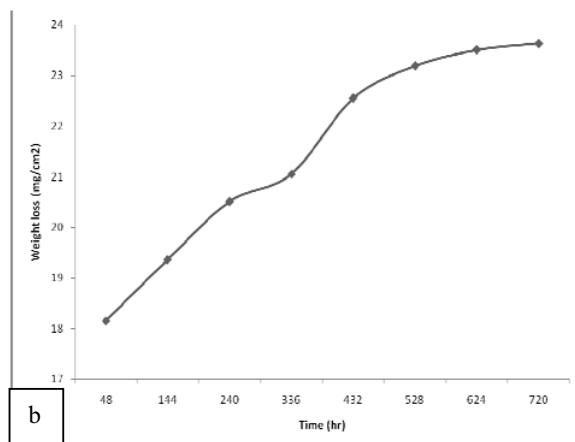
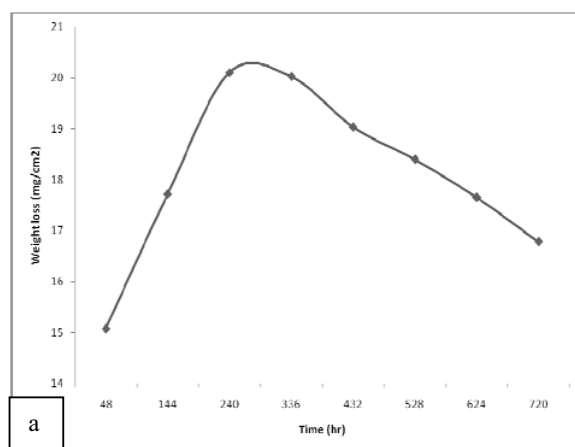
Energy Dispersive X-ray Spectroscopy (EDS) was used in conjunction with the Scanning Electron Microscope (SEM) JEOL, JSM-6420 to characterize the elemental composition of the sample after being immersed in sodium chloride solution.

Table 1 The composition (in weight percent) of the experimental Co-Cr-Ni alloys [5].

Sample no.	Composition (wt.%)
1	Co ₅₀ Cr ₅₀
2	Co ₅₅ Cr ₅ Ni ₄₀
3	Co ₅₇ Cr ₂₀ Ni ₂₃
4	Co ₆₁ Cr ₉ Ni ₃₀
5	Co ₆₂ Cr ₂₈ Ni ₁₀
6	Co ₆₈ Cr ₁₅ Ni ₁₇
7	Co ₇₃ Cr ₄ Ni ₂₃
8	Co ₇₅ Cr ₂₀ Ni ₅
9	Co ₈₁ Cr ₉ Ni ₁₀
10	Co ₉₁ Cr ₄ Ni ₅

III. RESULTS AND DISCUSSION

In order to study the corrosion rate of the Co-Cr-Ni alloys, an immersion test were conducted and the mass loss of samples were calculated using the data of an immersion test. Graph for weight loss (mg/cm²) vs. time (hour) were plotted [7] (Fig. 1) for all 10 samples. Overall, the graph plotted shows an increasing trend for weight loss as a function of time for all 9 samples of Co-Cr-Ni except for Co-Cr sample (Fig. 1 a) which show a decreasing trend of weight loss over time.



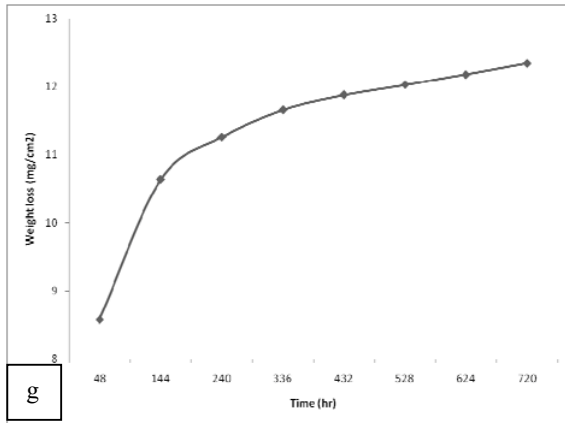
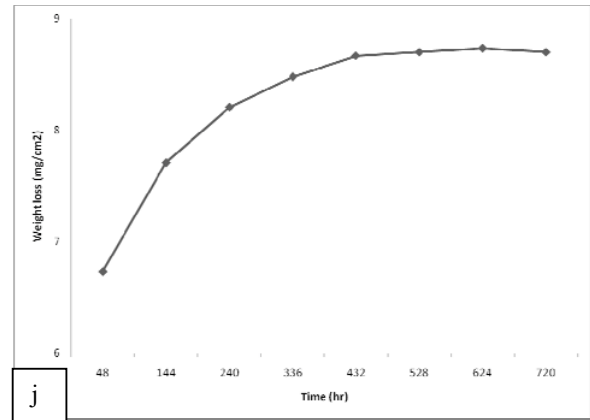
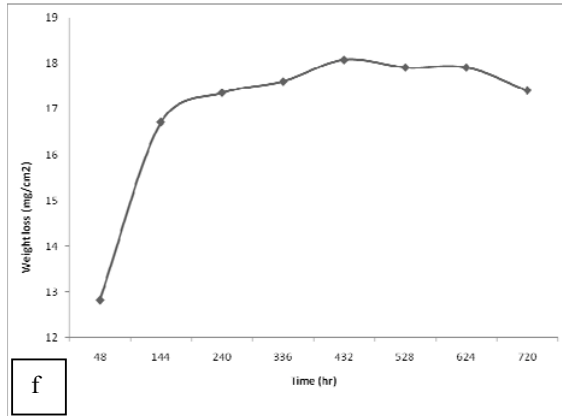
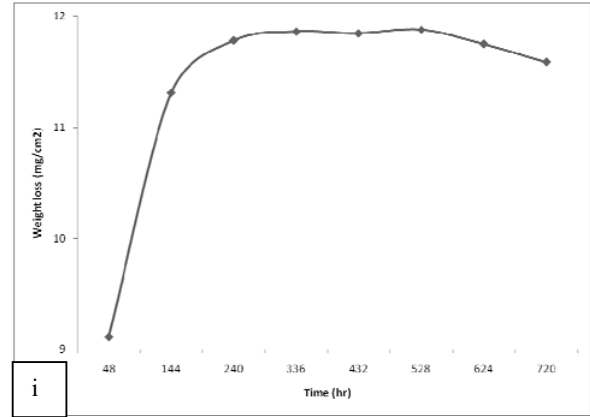
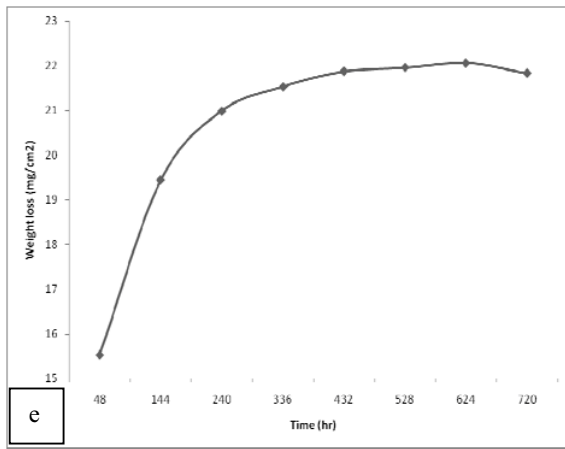


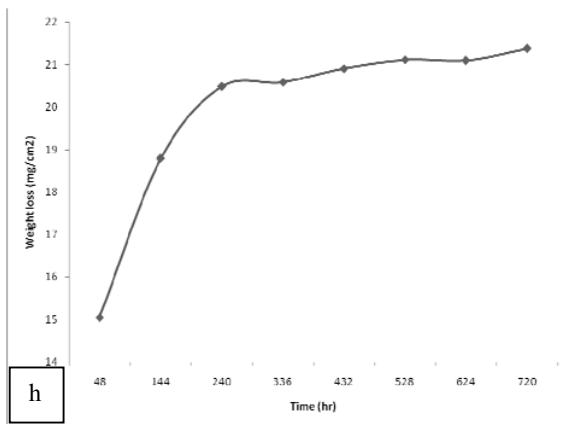
Fig. 1: Weight loss over time plot for a) $\text{Co}_{50}\text{Cr}_{50}$ b) $\text{Co}_{55}\text{Cr}_5\text{Ni}_{40}$ c) $\text{Co}_{57}\text{Cr}_{20}\text{Ni}_{23}$ d) $\text{Co}_{61}\text{Cr}_9\text{Ni}_{30}$ e) $\text{Co}_{62}\text{Cr}_{28}\text{Ni}_{10}$ f) $\text{Co}_{68}\text{Cr}_{15}\text{Ni}_{17}$ g) $\text{Co}_{73}\text{Cr}_4\text{Ni}_{23}$ h) $\text{Co}_{75}\text{Cr}_{20}\text{Ni}_5$ i) $\text{Co}_{81}\text{Cr}_9\text{Ni}_{10}$ j) $\text{Co}_{91}\text{Cr}_4\text{Ni}_5$

From the weight loss data, the corrosion rate using mils per year expression was applied using the following formula [8];

$$mpy = \frac{KW}{DAT}$$

where K is a constant, W is mass loss, g, D is density of specimen, g/cm^3 , A is area of specimen, cm^2 , T is time of exposure, hr. The calculated mpy is shown in Table 2.

Table 2: mpy results for 11 series of CoCrNi composition



composition	mpy (30days)
Co ₅₀ Cr ₅₀	14.32799776
Co ₅₅ Cr ₅ Ni ₄₀	16.76355657
Co ₅₇ Cr ₂₀ Ni ₂₃	17.25205325
Co ₆₁ Cr ₉ Ni ₃₀	14.86657304
Co ₆₂ Cr ₂₈ Ni ₁₀	17.06538637
Co ₆₈ Cr ₁₅ Ni ₁₇	12.55847158
Co ₇₃ Cr ₄ Ni ₂₃	8.732052432
Co ₇₅ Cr ₂₀ Ni ₅	16.7376707
Co ₈₁ Cr ₉ Ni ₁₀	7.868321193
Co ₉₁ Cr ₄ Ni ₅	5.744535205

Table 2 shows that sample Co₉₁Cr₄Ni₅ has the lowest mpy value which means this composition has the highest corrosion resistance than the other composition. This sample has the highest weight percent of Co and but the lowest weight percent of Cr among other composition. In contrary, sample with Co₅₇Cr₂₀Ni₂₃ composition recorded the highest mpy value, meaning that this sample has the lowest corrosion resistance among the others.

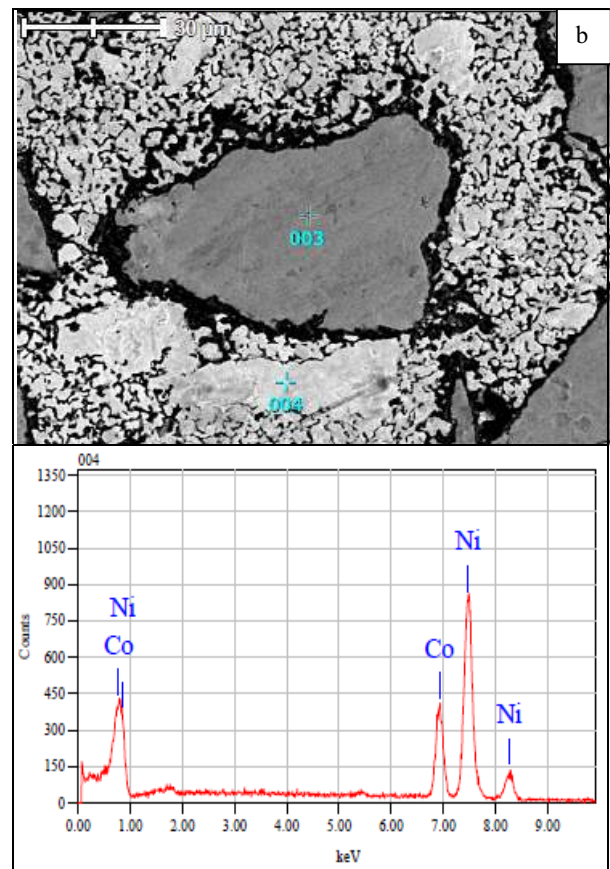
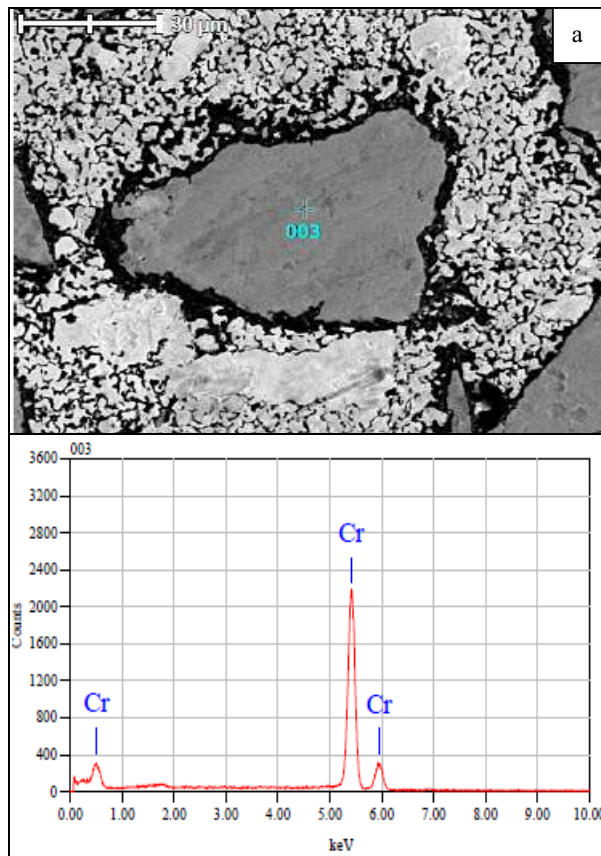


Fig. 2: EDS micrograph showing the element of a) Cr and b) Co and Ni after immersion test

EDS micrograph (Fig. 2) of the sample after the immersion test was taken to observe on how the corrosion attack the Co-Cr-Ni samples. From the observation made, EDS shows that the corrosion product was accumulated around the boundary of Cr that separated the region between Co-Ni and Cr phase in the micrograph.

IV. CONCLUSION

A study on corrosion of powder metallurgy Co-Cr-Ni alloy was performed in order to determine the optimal properties having the best corrosion resistance. The main results were as follows:

1. Sample with Co₉₁Cr₄Ni₅ composition has the lowest mpy result after 30 days immersed in 0.9% sodium chloride solution while sample with Co₅₇Cr₂₀Ni₂₃ composition recorded the highest mpy value.

2. an observation made through the EDS micrograph, corrosion product was accumulated around the boundary of Cr that separated the region between Co-Ni and Cr phase.

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