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Synthesis of Cobalt Alloy Through Smelting Method and Its Characterization as Prosthesis Bone Implant

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Abstract. Cobalt-based alloys are widely used as total hip and knee replacements because of their excellent properties, such as corrosion resistance, fatigue strength and biocompatibility. In this work, cobalt alloys with variation of Cr (28.5; 30; 31.5; 33, and 34.5% wt) have been synthesized by smelting method began with the process of compaction, followed by smelting process using Tri Arc Melting Furnace at 200A. Continued by homogenization process at recrystallization temperature (1250º C) for 3 hours to allow the atoms diffuses and transform into γ phase. The next process is rolling process which is accompanied by heating at 1200º C for ± 15 minutes and followed by quenching. This process is repeated until the obtained thickness of ± 1 mm. The evaluated material properties included microstructure, surface morphology, and hardness value. It was shown that microstructure of cobalt alloys with variation of Cr is dominant by γ phase, thus making the entire cobalt alloys have high hardness. It was also shown from the surface morphology of entire cobalt alloys sample indicated the whole process of synthesis that had good solubility were at flat surface area. Hardness value test showed all of cobalt alloys sample had high hardness, just variation of 33% Cr be in the range of ASTMF75, it were 345,24 VHN which is potential to be applied as an implant prosthesis.

Keywords: Cobalt alloys, prosthesis, microstructure, surface morphology, hardness.
PACS: 61.82.Bg, 61.10.Nz, 52.50.Gj, 62.20.Fe , 87.15.La, 87.15.By.

INTRODUCTION

Orthopedic surgery cases in Indonesia increased along with the number of old people, osteoporosis, osteoarthritis, and high rates of accident. Bone destruction especially of joint cartilage can be replaced with a prosthesis bone implant (biomaterial which is permanently implanted in the body about 15-20 years old) [1,2]. Fulfillment of prosthesis is still depending on the imports so that expensive and ineffective time for healing for the patient. Moreover, its size was not suitable for Indonesian people.

The main properties of biomaterials for implant prosthesis is its compatibility with living cells (excellent biocompatibility), no carcinogenics, durable and great strength of implant materials. It also must have good corrosion resistance and osseointegration [2-6].

Metal-based implants materials for prosthesis commonly use stainless steel, cobalt alloys, and titanium alloys [5,6]. Stainless steel has less corrosion resistance and if using titanium alloys is relatively expensive. While cobalt alloys have mechanical properties and biocompatibility better than stainless steel but lower than titanium alloys, and also the price is cheaper than titanium alloys [6].

Cobalt alloy Cr variations are expected improving the mechanical and non-mechanical properties of prosthesis bone implant. Cr is using for increasing the corrosion resistance by forming Cr_2O_3 passive layer and γ phase so that increased hardness of alloys [6,7].

Nowadays, there is no proper information surely of cobalt alloys composition that have mechanical and non-mechanical properties such as bones, especially for the femoral part. Of course it cannot be separated from the determination of the exact composition for cobalt alloys elements. Therefore, it is necessary to study more about the synthesis of a cobalt alloys prosthesis bone implant especially for the aspect of optimizing the Cr composition.

In this study, the synthesis of cobalt alloys with Cr variation refers to ASTMF75 composition. The variation of Cr were carried out at 28.5; 30; 31.5; 33, and 34.5% wt. Under appropriate standard of ASTMF75 another constant compositions are added by amount of 5% Mo, 0.5% Mn, 0.5% Si, 0,25% N, and the remaining Co (wt). Cobalt alloy metal manufacturing is done by smelting method and accompanied by rolling.

The aim of this study was to characterize the effect of Cr variations with smelting method were accompanied by rolling of the cobalt alloy. Expectation from this study were able to make cobalt alloy in accordance with the characterization of the prosthesis bone implant.

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EXPERIMENTAL PROCEDURE

The nominal compositions of Cr prepared in this study are 28,5%, 30%, 31,5%, 33%, and 34,5% (wt). Constantly component of this alloys are 5% Mo, 0,5% Mn, 0,5% Si, and 0,25% N (wt) and the remaining component is Co (TABLE 1). Experimental alloys were melted in Tri Arc Melting Furnace at 200A or equal about 3000ºC which was maintained under 3Pa of Ar (argon) gas pressure, previously began with the compacting for optimalization process. Continued by homogenization on recrystallization temperature about 1250 º C for 3 hours. Then, it entered rolling process which is accompanied by heating at 1200ºC about 15 minutes and followed by quenching. The process was repeated until the obtained thickness about 1 mm. Heating process was always in Ar atmosphere in order to decrease oxidation.

Characterization

Phase identification of the forged alloys was performed by X-ray diffractometer (XRD). X-ray diffraction patterns were measured between 2θ = 20º and 80º by Cu Kα radiation, then the step size of 2θ = 0,01. Phase identification is done three times, after smelting, homogenization, and rolling. Percentage of phase is calculated by the formula (1). Surface morphology and composition of the samples were identified by using SEM-EDX. Hardness test were performed with *Tester Digital Auto Turret model 402MVD.* Acquiring the average hardness test result, three different places of each specimens were measured.

TABLE 1. Cobalt alloy composition variations Cr

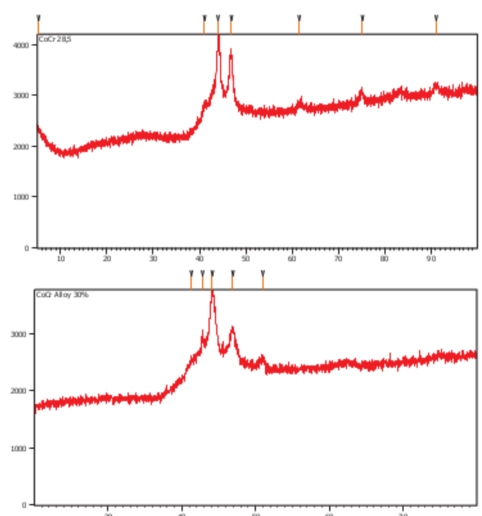
Variation of Cr composition (%)	composition (% wt)						Total weight (g)
	Cr	Mo	Mn	Si	N	Co	
28,5	28,5	5	0,5	0,5	0,25	65,25	10
30,0	30,0	5	0,5	0,5	0,25	63,75	
31,5	31,5	5	0,5	0,5	0,25	62,25	
33,0	33,0	5	0,5	0,5	0,25	60,75	
34,5	34,5	5	0,5	0,5	0,25	59,25	

$$\% phase_{\gamma,\varepsilon,\sigma} = \frac{\sum \text{intensity phase}_{\gamma,\varepsilon,\sigma}}{\sum \text{intensity phase}_{\gamma} + \sum \text{intensity phase}_{\varepsilon} + \sum \text{intensity phase}_{\sigma}} \quad (1)$$

RESULT AND DISCUSSION

Characterization results of XRD (X-Ray Diffraction)

FIGURE 1 shows diffractogram pattern of cobalt alloys after smelting. It is shown the success of smelting process which is indicated by the formation of a pattern that similar to the literature [7]. γ phase appears with the greatest intensity than another phase (θ and σ).



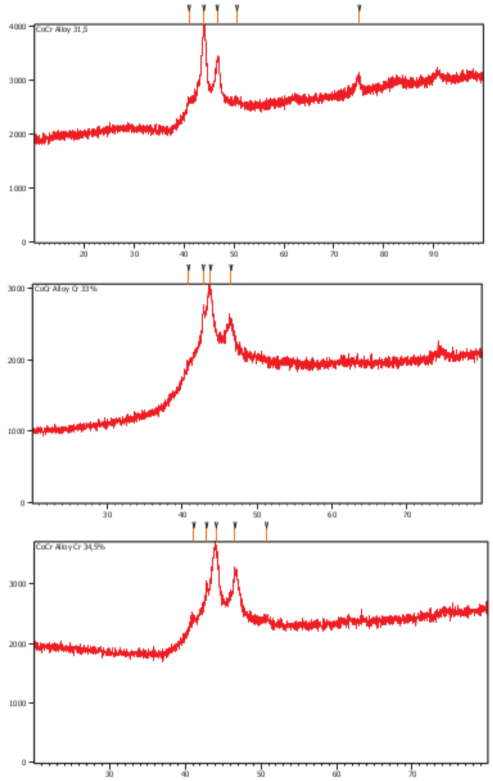
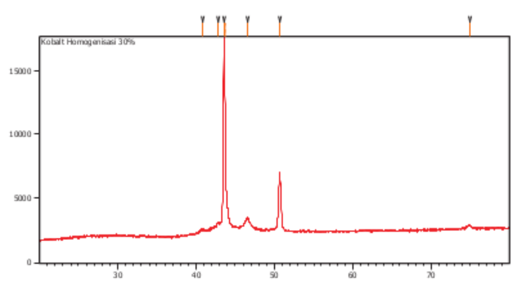


FIGURE 1. XRD Pattern of Cobalt Alloys after Smelting.

All phases of cobalt alloys appear because of allotropes properties. Intensity of γ phase is still quite low, so it is enhanced by homogenization at recrystallization temperature 1250ºC for 3 hours to allow the atoms to diffuse.



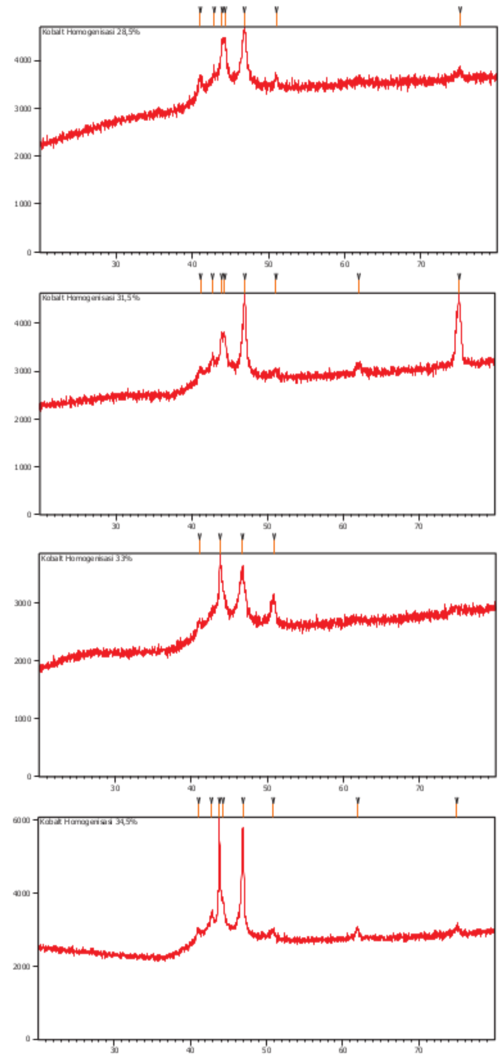


FIGURE 2. XRD Pattern of Cobalt Alloys after Homogenization.

FIGURE 2 shows the pattern of cobalt alloys after homogenization which is almost the same as the previous pattern that similar to the literature. It is also found the tapered pattern of the cobalt alloy because of the better crystallinity. Percentage of γ phase value also tends to increase when compared with after smelting process because of providing an opportunity for cobalt alloy atoms to transform to be γ phase.

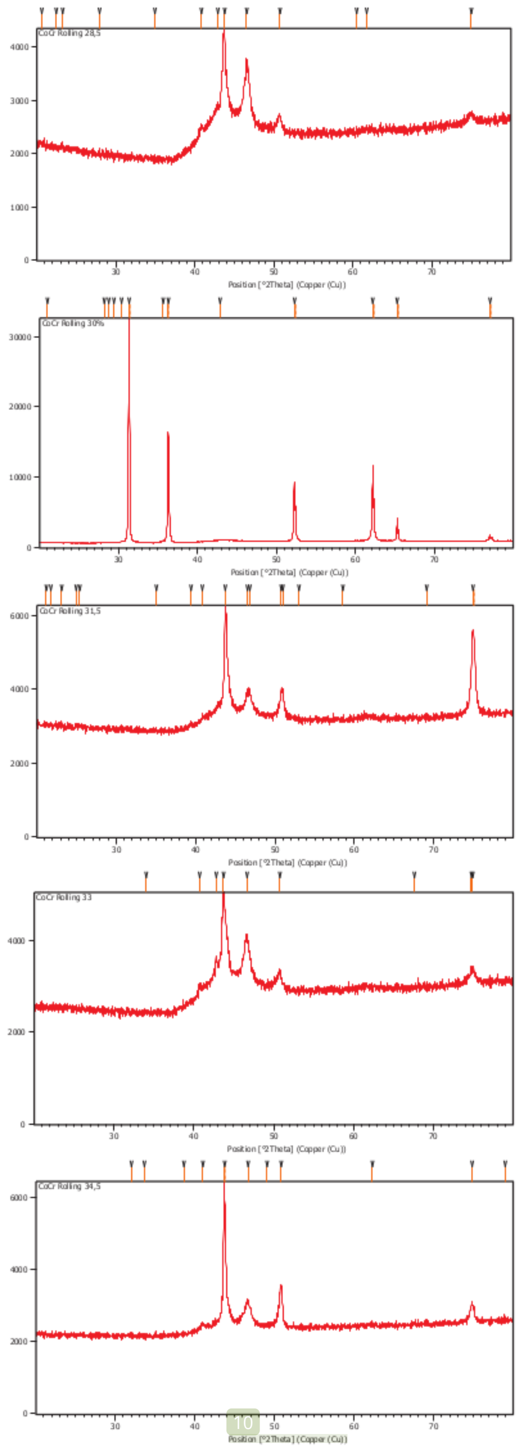


FIGURE 3. XRD Pattern of Cobalt Alloys after Rolling.

XRD analysis of the observations and cobalt alloys after rolling shows that there is increasing of crystallinity and percentage of the γ phase. This is because the rolling process that accompanied by the heating process carried out at a temperature of 1200ºC about 15 minutes. It process repeated until the thickness obtained. The function of heating is making the metal more malleable so that more easily the rolling process. It also provides an opportunity for ε and σ phase to be γ phase. Rolling process provides a substantial contribution becoming the γ phase, because the process is able to stored-strain energy, grain refining, and the inhibition of dislocation glide which was produced by carbide precipitation during hot forging so can optimize the appearance of the γ phase [8].

TABLE 2 shows the increase percentage of γ phase with increasing Cr variations, thereby increasing the hardness of cobalt alloys. The harder alloys makes easier the rolling process [8]. On the other hand, the rolling process gives a huge impact for the growth of γ phase. Therefore the increasing in Cr variation can increase γ phase.

TABLE 2. Identification the percentage of Cobalt Alloy Phase After Rolling.

Variation of Cr composition	γ phase (%)	ε phase (%)	σ phase (%)
28,5%	69,0	19,3	11,7
30,0%	0	c0,00	100
31,5%	79,8	0,00	20,2
33,0%	99,2	0,00	0,80
34,5%	96,5	0,00	3,50

Increase of γ phase from smelting to rolling shown on the FIGURE 4. Samples with a percentage of 30% Cr were not included because damaged.

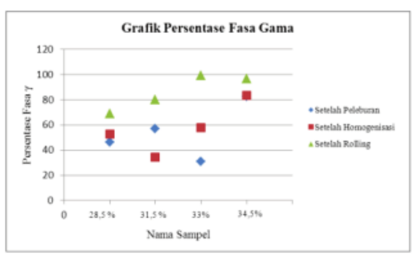
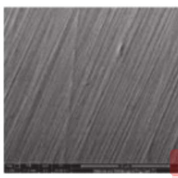


FIGURE 4. Percentage of γ phase from Smelting to Rolling.

Variation of 30% cobalt alloy Cr damaged after rolling through. It is because the γ phase does not appear at any angle of 2θ and the σ phase is the only one, so it caused brittleness material [7]. Physical observations indicate that the sample is more lenient than the others, the sample can be curved without tools while the other samples have relatively high hardness. The damage allegedly due to the melting process is not perfect, because incandescent plasma turned into such a burned wood when the smelting process occured. Melting process is not perfect so that make the bonding between the atoms is not perfect too.

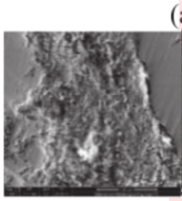
SEM-EDX Characterization

SEM-EDX characterization was conducted to know the surface morphology as well as the composition of the cobalt alloy elements contained in one point of sample, Figure 5-9 below shows the results of SEM-EDX of cobalt alloy surface after rolling taken at two different points, the normal area (flat surface) and the extreme area (Perforated). The aims of those points were to find differences or similarities characteristics of two areas that shooted by high-energy electron beam from the aspect of the alloys component elements appearance.



Element	Wt%	At%
MoL	06.09	03.68
CrK	28.93	32.29
MnK	00.61	00.65
CoK	64.37	63.38
Matrix	Correction	ZAF

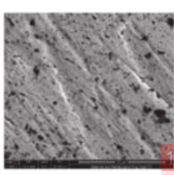
(a)



Element	Wt%	At%
OK	20.09	47.02
SiK	01.96	02.61
MoL	05.05	01.97
CrK	23.94	17.24
MnK	01.10	00.75
CoK	47.86	30.41
Matrix	Correction	ZAF

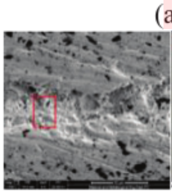
(b)

FIGURE 5. SEM-EDX Samples 28,5% (a) Flat Surface and (b) Perforated Surface



Element	Wt%	At%
OK	18.97	52.02
SiK	08.25	12.88
MoL	67.44	30.83
CrK	02.29	01.93
MnK	01.11	00.88
CoK	01.95	01.45
Matrix	Correction	ZAF

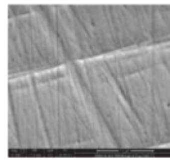
(a)



Element	Wt%	At%
OK	16.04	44.16
SiK	13.54	21.25
MoL	63.65	29.23
CrK	01.98	01.68
MnK	01.98	01.59
CoK	02.81	02.10
Matrix	Correction	ZAF

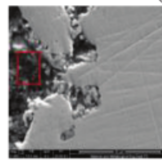
(b)

FIGURE 6. SEM-EDX Samples 30,0% (a) Flat Surface and (b) Perforated Surface



Element	Wt%	At%
OK	04.94	15.76
SiK	00.42	00.76
MoL	06.20	03.30
CrK	30.21	29.67
MnK	00.54	00.50
CoK	57.70	50.01
Matrix	Correction	ZAF

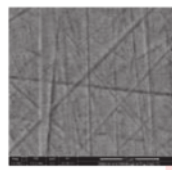
(a)



Element	Wt%	At%
OK	25.85	50.80
SiK	14.39	16.11
MoL	03.53	01.16
CrK	26.17	15.83
MnK	01.97	01.13
CoK	28.09	14.99
Matrix	Correction	ZAF

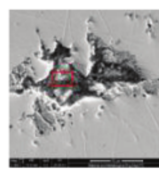
(b)

FIGURE 7. SEM-EDX Samples 31,5% (a) Flat Surface and (b) Perforated Surface



Element	Wt%	At%
OK	05.53	17.42
SiK	00.94	01.68
MoL	06.78	03.56
CrK	27.94	27.06
MnK	00.65	00.60
CoK	58.15	49.68
Matrix	Correction	ZAF

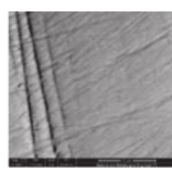
(a)



Element	Wt%	At%
OK	31.83	60.99
SiK	01.32	01.44
MoL	01.79	00.57
CrK	44.77	26.39
MnK	01.47	00.82
CoK	18.83	09.79
Matrix	Correction	ZAF

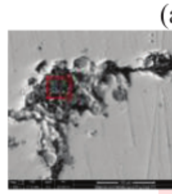
(b)

FIGURE 8. SEM-EDX Samples 33,0% (a) Flat Surface and (b) Perforated Surface



Element	Wt%	At%
OK	05.14	16.24
SiK	00.75	01.35
MoL	06.45	03.40
CrK	32.89	31.99
MnK	00.56	00.51
CoK	54.20	46.50
Matrix	Correction	ZAF

(a)



Element	Wt%	At%
OK	37.26	57.57
SiK	34.81	30.64
MoL	03.11	00.80
CrK	10.04	04.77
MnK	00.45	00.20
CoK	14.33	06.01
Matrix	Correction	ZAF

(b)

FIGURE 9. SEM-EDX Samples 34,5% (a) Flat Surface and (b) Perforated Surface

Based on EDX analysis which is shown in Figure 5-9, cobalt alloys with a flat surface have the same weight percentage ratio of alloying elements with weight percentage when measured, although the weight percentage of the alloying elements decreased. Whereas the perforated surface undergoes oxidation, it is alleged by the existence of O elements in EDX analysis.

The O elements interact with one of the passive layer compound cobalt alloys so make oxidizing. Direct physical observations indicate that the oxidized part are colored black. In addition, by SEM-EDX observations perforated area only consist of one or a few components.

Microvickers Hardness Characterization

In this study the tested mechanical properties were hardness samples. Characterization results of hardness tests are shown in the Table 3 below.

TABLE 3. The Result of Value Hardness Number (VHN) of cobalt alloys

Variation of Cr composition	Hardness *(VHN)*
28,5%	537,0
31,5%	556,0
33,0%	345,3
34,5%	419,4

Cobalt alloys with increasing Cr variation should increase the hardness value. But the result is differ from the expectation. The entire sample has a high hardness. Cobalt alloy hardness is quite high due to Cr can substitute the Co atom. Substitution occurs because of the difference in size between them is quite small. Cr atomic number (24) is smaller than the Co (27) so that the radius of Cr is smaller than the Co radius. Therefore more and more atoms of Cr are added to the alloy, Cr atoms are substituted so make the alloy material hardness even great.

The results are obtained different as expected. Hardness value of the samples exceeded the average of the range for cobalt alloys implants accordance to ASTMF7 (276 to 384.6 *VHN*). The best result of hardness test is the variation of Cr 33%, that is 345.24 *VHN*.

Conclusions

In order to improve the mechanical properties of cobalt alloys, we have examined the effect of Cr variation based on microstructure and mechanical properties of the cobalt alloys by rolling process. Because Cr is able to increase the growth of γ phase so it can increase the hardness value of alloys. Hardness value of the entire samples had very high of hardness, however this value is possible implanted in the body. Thus the present study shows that the cobalt alloys with Cr variation up to 33 mass% is potential to be applied as an implant prosthesis.

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