Characterization of Co-Cr-Mo (F-75) alloy produced by solid state sintering

Zuraidawani Che Daud, Shamsul Baharin Jamaludin and Fazlul Bari

Abstract
This research was carried out to fabricate and characterize Co-Cr-Mo (F-75) alloy. The samples have been prepared via solid state sintering. The powders were supplied by Sandvik Osprey Ltd, UK and special design for biomedical applications. The lab work comprises the mixing of F-75 powder with 2 wt. % of binder. The mixture was cold compacted using uniaxially press at 500 MPa. The samples were sintered at three different temperatures (1250 °C, 1300 °C and 1350 °C) in inert environment for 90 minutes of sintering time. The sintered samples were characterized by using scanning electron microscope (SEM), energy dispersive X-ray spectroscopy (EDS) and optical microscope (OM) *Olympus BX41M*. Bulk density, apparent porosity, percentage of linear shrinkage, and microhardness of the samples were also characterized. The average of the grain sizes were measured by line intercepts method. The optical micrographs showed the difference grain size in all sintered samples after etching with Marble reagent. The result shows the percentage of linear shrinkage, bulk density value and porosity increase with increasing the sintering temperature. Beside that, higher sintering temperature yields coarser grain structure.

Keywords: Co-Cr-Mo alloys, physical properties, solid state sintering

Zuraidawani Che Daud. Author is with the School of Material Engineering, University Malaysia Perlis, Perlis, Malaysia. (phone no: +60124899314; fax: +6049798178; e-mail:wanie278@gmail.com).

Shamsul Baharin Jamaludin. Author is with the School of Material Engineering, University Malaysia Perlis, Perlis, Malaysia. (e-mail: sbaharin@unimap.edu.my).

Fazlul Bari. Author is with the School of Material Engineering, University Malaysia Perlis, Perlis, Malaysia. (e-mail: fazlul@unimap.edu.my).

I. INTRODUCTION

For generation various materials so-called biomaterials are used in medicine and dentistry with a purpose to replace or repair a body feature, tissue, organ or function. The performance of biomaterial in direct contact with living tissue is controlled by two sets of characteristics; biofunctionality and biocompatibility [1]. The main metallic biomaterials are stainless steels, Co-based alloys and titanium and its alloys. These materials have been attracted much interest for their use as medical implants such as hips, knees, bone plates, ankles and dental implants [2-4]. Among the metallic materials, Co-Cr-Mo (F-75) alloys exhibit the most useful balance in strength, fatigue, good wear and corrosion resistance as well as biocompatibility [5, 6].

The most of Co-Cr-Mo implants have been manufactured using casting technique. Casting technique has provided desirable processing flexibility and lower initial costs. However, distinct limitations have been associated with casting such as coarse grain size, non-uniform microstructural segregation and also lower tensile and fatigue strength [7]. Then the fabrications were improved using hot forging and powder metallurgy method. Powder metallurgy (P/M) Co-Cr-Mo alloy offers certain unique options for the design and fabrication of surgical implants intended as permanent tissue replacements [8].

The study of P/M for biocompatible materials (Cobalt F-75) is less compared to another process. Because of that, P/M method was used in this research to improve process for metal implant instead of casting. Furthermore, through P/M method the near net shape part will produce easily. Conventionally, P/M process consists of mixing, compaction and sintering.

Solid state sintering is the most common technique for consolidating powders. Essentially, it is the removal of the pores between the starting particles, combined with their growth and strong mutual bonding. The process is carried out by heating up the green body at about 80 % of the melting temperature, until full strength is achieved (10min to several hours) [9, 10]. Reference [11] have studied the sintering of biocompatible P/M Co-Cr-Mo alloy (F-75) for fabrication of porosity graded composite structures. They investigated the different of sintering temperature starting 1280 °C up to 1360 °C for 120 min of sintering time. Their result showed that sintering F-75 alloy in argon atmosphere gave the highest corrosion resistance compared with those sintered in vacuum and $75H_2$-$25N_2$ atmosphere. Recently, the P/M technology has been used to produce a porous Co-Cr-Mo based composite material with the bioactive glass addition and it was studied by [12]. They used a water atomised pre-alloyed F-75 powder mixed with bioglass, compact and then sintered at 1230 °C for 1 hour in argon atmosphere. They found that the addition of bioglass to the matrix of F-75 alloy, as well as rotary cold repressing and heat treatment of sintered specimens have changed the microstructure, mechanical and corrosion properties of composite materials. This research has focused on the effect of sintering temperature on the microstructure and physical properties of Co-Cr-Mo (F-75).

II. EXPERIMENTAL PROCEDURE

Co-Cr-Mo alloy powder was used in this research. This powder was produced by gas atomization with the chemical composition according to ASTM F 75. The alloy powders were supplied by Sandvik Osprey Ltd, UK and special design for biomedical applications. Table 1 shows the chemical compositions of the powder.

Table 1: Chemical composition (wt. %) of Co-Cr-Mo (F 75) powders

Element	Co	Cr	Mo	Si	Mn	Ni	Fe	C
Weight percent (wt. %)	62.595	29.3	6.1	0.79	0.74	0.26	0.20	0.015

Particle size distribution of the powder was determined by using Malvern particle size analyzer. Figure 1 shows the SEM micrograph of the powder particles, exhibiting nearly spherical shape of particles. The average size of Co-Cr-Mo particles is about 8.8 µm

The samples were prepared by powder metallurgy method. The powder and 2 wt. % of stearic acid as a binder were blended for 30 min to prevent segregation due to free-fall and vibration during mixing. The mixed powder was poured into a die with 13 mm diameter and compacted using uni-axially cold press at a pressure of 500 MPa. The green compact was sintered using WEBB 84 furnace at three different temperatures (1250 °C, 1300 °C and 1350 °C) for 90 minute of sintering time with 10 °C/min heating rate in inert atmosphere.

The percentage of linear shrinkage of each sample was determined by using (1);

$$\text{Linear shrinkage (\%)} = \frac{l_o - l_f}{l_o} \times 100 \qquad (1)$$

Where;

l_o = initial dimension of green body (mm).

l_f = final dimension of sintered sample (mm).

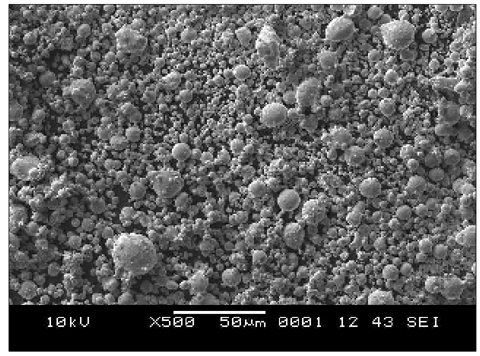

Figure 1: SEM micrograph of the Co-Cr-Mo alloy particles.

Bulk density and apparent porosity value of each sintered samples were obtained by Archimedes method according to ASTM B311-93 standard [13]. The bulk density and percentage of apparent porosity were calculated by using equation (2) and (3) respectively.

$$\text{Bulk density}\left(\text{g}/\text{cm}^3\right) = \frac{W_a}{W_c - W_b} \times \rho_{water} \qquad (2)$$

$$\text{Apparent porosity (\%)} = \frac{W_c - W_a}{W_c - W_b} \times 100 \qquad (3)$$

Where;

W_a = mass of sample in air (g).

W_b = apparent mass of test sample (g).

W_c = saturated mass of test sample (g).

The microstructures of the sample were studied under dark field of optical microscope (Olympus BX41M) and Scanning Electron Microscope (JSM-6460LA). The average of grain size was measured by using line intercepts method. Vickers microhardness measurements were carried out on the polished surface sample by using HM-114 Mitutoyo Hardness Testing Machine. The compression tests were carried out according to ASTM standard E9 at 0.5 mm per minutes.

III. RESULTS AND DISCUSSION

Table 2 shows the sample characteristics of sintered sample at three different sintering temperatures. The overall result shows the percentage of shrinkage, bulk density value, porosity and grain size were increased with increasing the sintering temperature. Meanwhile, the compressive strength decreases with increasing the sintering temperatures. Obviously, sintering strongly affects the final density, grain size and mechanical properties of the sample [14]. It can be seen the microhardness values are 315, 293, and 318 HVN for 1250 °C, 1300 °C and 1350 °C respectively.

Table 2: Sample characteristics of F-75 alloy after sintered at different temperatures.

Sample Characteristics	Temperature (°C)		
	1250	**1300**	**1350**
Shrinkage (%)	9.87	11.74	14.76
Bulk density (g/cm^3)	7.09	7.23	7.49
Apparent porosity (%)	1.96	1.90	0.02
Grain size (µm)	46.7	53.0	87.0
Compressive strength (MPa)	1375	298	255
Microhardness (HVN)	315	293	318

Referring to the Figures 2(a) to 2(c), micrograph shows the F-75 sintered sample at different sintering temperatures. From the observation, it is seen that the small grains are embedded in the large grains. It also shows that, at temperature 1250 °C and 1300 °C, the pores located within the grain and at the grain boundary. Meanwhile, at higher temperature, 1350 °C most of the pores located at the grain boundary. Figures 3(a) to 3(c) show the SEM micrographs of F-75 sintered sample at three different temperatures meanwhile, Figures 3(d) to 3(f) represent the EDS pattern for overall image analysis. Overall image analysis showed the existence element of Co, Cr, Mo and Si.

In this study, solid state diffusion plays a major role in the formation and growth of interparticle bonding, thus diffusion bonding has a major effect on microstructure and mechanical properties. Diffusion itself is dependent on sintering temperature [15]. Based on micrographs, by increasing the sintering temperatures the pore morphology has changed to spherical pores within the grains or located at the grain boundaries.

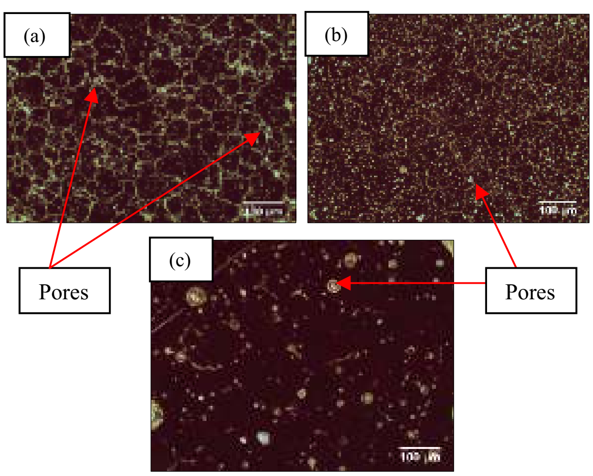

Figure 2: Optical micrographs of F-75 sintered sample at different sintering temperature. (a) 1250 °C, (b) 1300 °C and (c) 1350 °C.

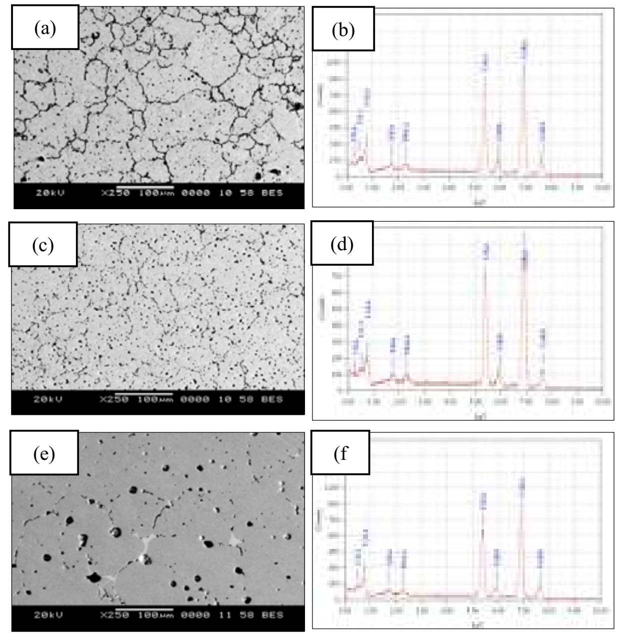

Figure 3: (a)-(c) SEM micrographs of sintered sample at different sintering temperature (1250 °C, 1300 °C and 1350 °C). (d)-(f) EDS pattern for sintered sample at different sintering temperature (1250 °C, 1300 °C and 1350 °C).

IV. Conclusions

1. The Co-Cr-Mo (F-75) alloy powder has been successfully fabricated by solid state sintering.
2. The different of sintering temperatures has affected the microstructures and physical properties of the sintered sample.
3. The percentage of linear shrinkage, bulk density, porosity and grain size increase with increasing the sintering temperature.
4. By increasing the sintering temperatures, the pore morphology has changed to spherical pores within the grain and located at the grain boundaries. Beside that, small grain was observed in the large grains.

Acknowledgment

This study has been supported by the science fund grant No. 9005-00008.

REFERENCES

[1] T. Matković, P. Matković, and J. Malina, "Effects of Ni and Mo on the microstructure and some other properties of Co-Cr dental alloys," *Journal of Alloys and Compounds,* vol. 366, pp. 293-297, 2004.

[2] D. H. Kohn, "Metals in medical applications," *Current Opinion in Solid State and Materials Science,* vol. 3, pp. 309-316, 1998.

[3] A. Marti, "Cobalt-base alloys used in bone surgeryKobalt-Basislegierungen in den knochenchirurgieAlliages à base de cobalt utilisés en chirurgie osseuseAleaciones con base de cobalto utilizadas en cirugía ósea," *Injury,* vol. 31, pp. D18-D21, 2000.

[4] M. Niinomi, "Recent metallic materials for biomedical applications," *Metallurgical and Materials Transactions A,* vol. 33, pp. 477-486, 2002.

[5] D. Sheeja, B. K. Tay, S. P. Lau, and L. N. Nung, "Tribological characterisation of diamond-like carbon coatings on Co-Cr-Mo alloy for orthopaedic applications," *Surface and Coatings Technology,* vol. 146-147, pp. 410-416, 2001.

[6] S. Spriano, E. VernÃ¨, M. G. Faga, S. Bugliosi, and G. Maina, "Surface treatment on an implant cobalt alloy for high biocompatibility and wear resistance," *Wear,* vol. 259, pp. 919-925, 2005.

[7] M. J. Walter, "Benefits of P/M Processed Cobalt-Based Alloy for Orthopaedic Medical Implants ": Carpenter Technology Corp., Wyomissing, USA 2006.

[8] R. M. Pilliar, "Porous P/M structures for orthopaedic and dental implant applications," *International Journal of Powder Metallurgy (Princeton, New Jersey),* vol. 40, pp. 19-27, 2004.

[9] A. K. Eksi and A. H. Yuzbasioglu, "Effect of sintering and pressing parameters on the densification of cold isostatically pressed Al and Fe powders," *Materials and Design,* vol. 28, pp. 1364-1368, 2007.

[10] R. M. German, *Sintering Theory and Practice*. New York, NY: John Wiley & Sons, INC., 1996.

[11] M. Dourandish, D. Godlinski, A. Simchi, and V. Firouzdor, "Sintering of biocompatible P/M Co-Cr-Mo alloy (F-75) for fabrication of porosity-graded composite structures," *Materials Science and Engineering: A,* vol. 472, pp. 338-346, 2008.

[12] Z. Oksiuta, J. R. Dabrowski, and A. Olszyna, "Co-Cr-Mo-based composite reinforced with bioactive glass," *Journal of Materials Processing Technology,* vol. 209, pp. 978-985, 2009.

[13] B311-93, "Metallic and inorganic coating; Metal powder, sintered P/M structural parts," in *Annual book of ASTM standard* USA, 1999.

[14] E. Salahinejad, R. Amini, M. Marasi, and M. J. Hadianfard, "The effect of sintering time on the densification and mechanical properties of a mechanically alloyed Cr-Mn-N stainless steel," *Materials & Design,* vol. In Press, Corrected Proof, 2010.

[15] X. Xu, P. Lu, and R. M. German, "Densification and strength evolution in solid-state sintering. Part II. Strength model," *Journal of Materials Science,* vol. 37, pp. 117-126, 2002b.