

PAPER • OPEN ACCESS

Effect of sintering temperature on physical properties & hardness of CoCrMo alloys fabricated by metal injection moulding process

To cite this article: Ahmad Ridhwan Abdullah *et al* 2017 *IOP Conf. Ser.: Mater. Sci. Eng.* **257** 012010

View the [article online](#) for updates and enhancements.

Related content

- [Investigation of mechanical properties for open cellular structure CoCrMo alloy fabricated by selective laser melting process](#)
A Azidin, Z A M Taib, W S W Harun et al.
- [Paraffin wax removal from metal injection moulded cocrmo alloy compact by solvent debinding process](#)
N A N Dandang, W S W Harun, N Z Khalil et al.
- [Effect of sintering temperature on structural defects and superconducting properties in \$MgB_2 + C_4H_8O_2\$](#)
M S A Hossain, J H Kim, X Xu et al.

Effect of sintering temperature on physical properties & hardness of CoCrMo alloys fabricated by metal injection moulding process

Ahmad Ridhwan Abdullah¹, Nur Aidah Nabihah Dandang², Nur Zalikha Khalil³, Wan Sharuzi Wan Harun³

¹Mechanical Engineering Department, Politeknik Sultan Haji Ahmad Shah (POLISAS), Jalan Semambu, Pahang, 25350 Kuantan, Malaysia.

²Institute of Postgraduate Studies, Universiti Malaysia Pahang, Lebuhraya Tun Razak, 26300 Gambang, Kuantan, Pahang Malaysia.

³Green Research for Advanced Materials Laboratory, Human Engineering Group, Faculty of Mechanical Engineering, Universiti Malaysia Pahang, 26600 Pekan, Pahang, Malaysia.

*Corresponding author: ridhwan@polisas.edu.my

Abstract. Metal Injection Moulding (MIM) process is one of the Powder Metallurgy manufacturing techniques utilised to produce Cobalt Chromium Molybdenum (CoCrMo) compacts. The objective of this study is to determine physical properties and hardness of CoCrMo alloy compact sintered at three different sintering temperature at the similar soaking time. At the beginning, sample were fabricated by using Injection Moulding machine. Cobalt Chrome Molybdenum (CoCrMo) metal powder was selected for this study. A morphological study was conducted using optical microscope (OM) and micro-Vickers hardness testing. From the result obtained, it shows upward trend either on the hardness or physical properties of the samples. CoCrMo sintered compact become harder and volume of pores on surface become less due to the increase on sintering temperature. However, effect of increasing sintering temperature shows significant shrinkage of the sample, beginning losses in dimensional accuracy. It is discovered that a little change in sintering temperature gives significant impact on the microstructure, physical, mechanical of the alloy.

1. Introduction

The CoCrMo alloys that containing 28% weight of Chromium, 6% weight of Molybdenum (balance Co) [1] is one of the important materials in the biomedical field, particularly in the manufacture of body implant and dental surgery. The advantages of this material are durable, high mechanical strength and resistant to rust. This alloy has a resistance to corrosion due to the presence of chromium in the material. Molybdenum also serves to increase the strength of the alloy [2-4]. But, in any cases, unfavourable side effect like allergic cause by phenomenon called 'metallosis' commonly happen in biomedical field[5]. This situation makes the material less used in industry. Therefore, there is opportunities for researchers to study this material further in order to improve their mechanical properties.

Compacts from CoCrMo could be processed by using at least three techniques; powder metallurgy, casting, and hot forging. Powder metallurgy provide attractive resolution in order to explore and study



new material [6]. Well organized open cavity or porosity and probability of fabricating any materials are the advantages of Powder Metallurgy methods. Some examples of techniques available in Powder Metallurgy are Metal Injection Moulding (MIM) and Selective Laser Melting (SLM).

Metal injection moulding (MIM) is an innovation technique, savvy financial process for large scale manufacturing for produce complex, accurate, net shape parts or component by using either metal powder or clay powder. Capability of MIM depends on capacity to adapt complexity of design of plastic injection moulding and infinite selection of material offered, make it potential to merge with several parts to become solitary significant component [7, 8]. Internal crack in casting, defect cause by tolerance error, capital gain in casting manufacturing, and shape limitation are common problems that been faced by industries that easily can be solved by implementing MIM [9, 10]. MIM production is conquered by automotive sectors, particularly in Europe. Meanwhile, in the USA, focus for MIM is in biomedical/ healthcare. Instead, in Asia region, application of MIM is more to electronics and information technology field. Also, another growing factor is the development in the manufacturing of medical tools in Asia region, known as a greater population gains access to enhanced health care [11].

Basically, to improve mechanical properties, there are several methods that been used by researchers such as Severe Plastic Deformation (SPD). This method had been used to induce high strain into metallic materials. CoCrMo that been induced with high strain can cause the development of grain refinement. It is conceivable to refine grain size of cobalt in range 10-20 nm, heading to sharply increment up to 5 times in hardness and tensile strength [12]. Pure Cobalt (Co) recorded ultimate tensile strength and hardness normally around 210 MPa and 1700 MPa, and can be reached up to 750 MPa and 3530 MPa after grain refinement achieved [13]. In the other words, because of grain refinement, it improves mechanical properties of the material as well. Bolton and Becker [14] in their research have studied the effects sintering atmosphere on physical properties of CoCrMo alloys for surgical implants. It shown that by changing or manipulated certain parameters, level of porosity on materials could be changed either high or low. Compared sintering of CoCrMo alloy in argon with vacuum atmosphere shows that it produces high corrosion resistance. Densification and microstructure growth of CoCrMo during sintering were studied by many researchers to evaluate the compatibility of dissimilar grain sizes for co-sintering. Two components must have the same sintering behaviour to decrease gap stress during heating and cooling cycles. A study had been conducted by Heaney et al [15] to determine effect of sintering condition to final product of CoCrMo by using MIM. They stated that in the case of liquid phase sintering, the materials must have approximately 5% or less difference in shrinkage at or near the prescribed sintering temperature.

The objective of this present work is to study the effect of different sintering temperature and constant soaking time on the microstructure, physical properties, and hardness of CoCrMo alloy compact fabricated by metal injection moulding process.

2. Experimental Procedure

Table 1 representing the composition of raw material of CoCrMo. The CoCrMo alloy powder supplied from industry was fabricated through water atomization. Figure 2 shows Particle's profile is in spherical shape. Table 2 shows sample conditions involved in the study. Metal powder were mixed with polymer binder that consisted of Paraffin Wax, Polypropylene, and Stearic Acid to produce feedstock. Mixing ratio selected for feedstock is 65 volume % metal powder and 35 volume % binder system.

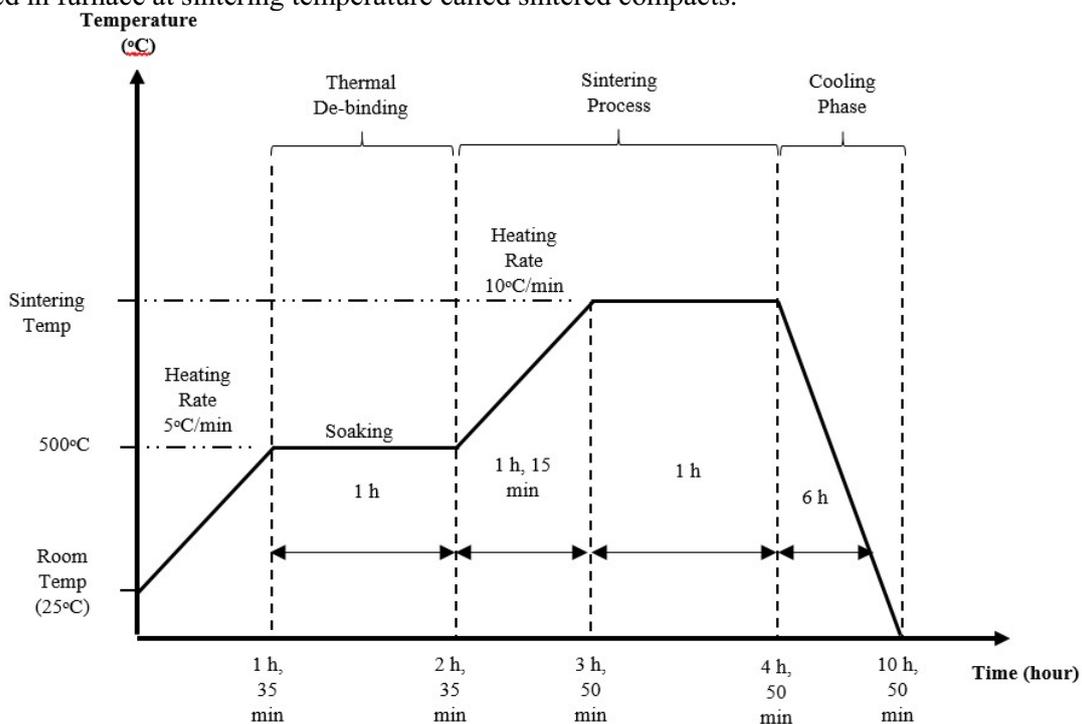
Table 1. CoCrMo alloy powder composition

Composition	Co	C	Cr	Mo	Ni	Mn	Si	Fe
Volume (%)	Balance	0.4	28.5	6.0	1.0	1.0	1.0	0.8

Table 2. The sample conditions

Sintering			Type of Metal Powder	Type of Binder	Mixing Ratio
Temperature	Time	Atmosphere			
1250 °C	2 hours	High purity Argon gas flow	Cobalt Chrome	Paraffin Wax,	65 % metal powder + 35 % binder
1300 °C			Molybdenum (CoCrMo)	Polypropylene, and Stearic Acid	
1350 °C					

Cylindrical samples with 20 mm diameter and 10 mm thickness were produced by using Metal Injection Moulding (MIM), equipped by standard moulding equipment under high pressure unit. Moulded part is referred to as a 'green compact'. Meanwhile, solvent de-binding is done by submerging green part in gaseous ethanol. This process had been done in closed container. Heat supplied by heater and temperature of the container was set at 60 °C. Next, compacts were put in a tube furnace in atmosphere of argon. Thermal de-binding and sintering was implemented at temperature of 1250, 1300 and 1300 °C separately. Figure 1 shows the temperature pattern for de-binding and sintering process. De-binding process is initiated by heating green compacts from room temperature to 500 °C with heating rate at 5 °C/min, takes at least one and a half hours. Then, compacts left exposed at temperature 500 °C for another one hour. This process is called 'Soaking'. Soaking process is to remove residual binder remaining in the compact. Then, furnace temperature was raised at rate 10 °C/min until it reaches the desired sintering temperature (1250, 1300, and 1350°C) respectively. Sintering process is carried out at constant temperature for one hour. Finally, compact is allowed to cool slowly in over six hours in furnace after shutdown. Sample that been burned in furnace at sintering temperature called sintered compacts.

**Figure 1.** Temperature pattern for de-binding and sintering

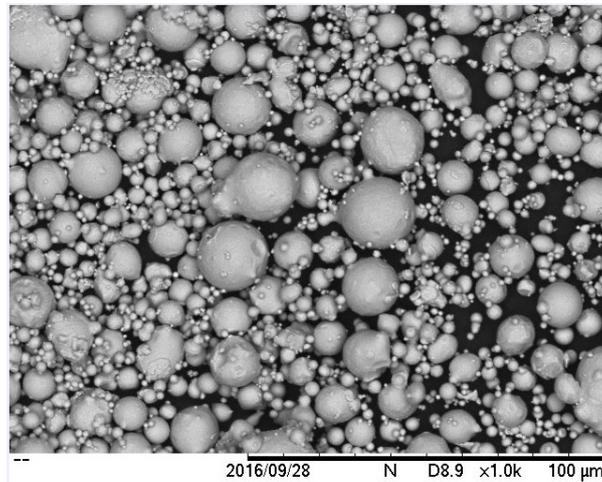


Figure 2. Spherical shape of CoCrMo particles

Sintered compacts were analysed by using optical microscope (OM) to determine porosity level via microstructure image. Correlation between microstructure result and mechanical properties of the sample were investigated via hardness test by using Vickers Micro Harness Machine.

3. Results and discussion

3.1 Microstructure

Figure 3 describe the sintered and polished compacts without etching with chemical reagent to expose the level of porosity. Analysis of the porosity level reveal that CoCrMo sintered compact which burned in 1350°C showed the lowest amount of pore. Meanwhile, sintered compact at sintering temperature 1250°C showed huge volume of porosity or open cavity as well. Forming of porosity on compact began in solvent de-binding process. Pores on the compacts surface actually becomes passage way for remaining binder left out from compact during thermal de-binding. During sintering process, exposure to high temperature causing amount of pores shrink. High temperature when performing the sintering procedure diminished pores on the surface of the parts. It obviously as a result of the recrystallization procedure of the metal particles, where the molecule of metal bonded each other together as inter-particle necks grow during sintering and porosity level is reduce[4, 16, 17]. When sintering process remains at higher temperature, the residual porosity in the compact parts reduces because pores are eliminated by bulk diffusion to grain boundaries, which contributes to an increase in relative density of the powder compact. Besides, high melting point temperature of CoCrMo powder lead to a diffusion, which porosity removed by increasing the sintering temperature [2, 3, 10].

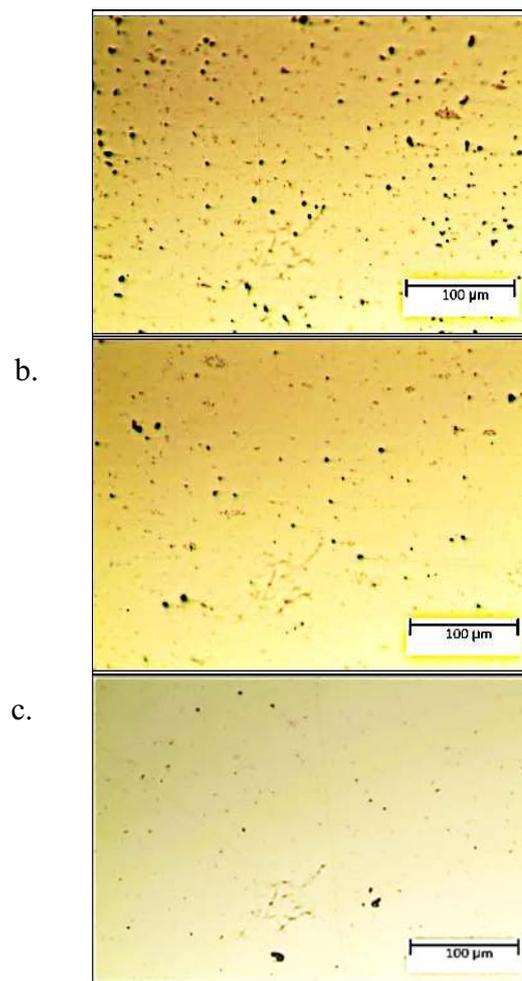


Figure 3. Amount of porosity for sintered compacts; sintering temperature; a) 1250 °C; b) 1300 °C; and c) 1350 °C

Shrinkage during solidification is the amount transformation of iron or metal in transitory from the melted state to the hard state at the freezing temperature, or the transition in volume of an alloy in passing from the liquid state at the beginning of the freezing range to the solid state at the end of the freezing range. The shrinkage undergone during solidification of an inter-metallic compound, freezing at a constant temperature, is similar to that of a pure metal. In figure 4, sintering temperature 1250 °C recorded the lowest shrinkage rate. With the increasing of sintering temperature, shrinkage rate also increased. So, at the highest temperature 1350°C, sintered compacts recorded the highest shrinkage rate. Figure 5 shown the decreasing or beginning loss in dimensional accuracy. At high temperature 1350°C, the sintered part showed lower than 73% similarity with eventual desired product specification. Component become increasingly smaller and relatively far away from net shape.

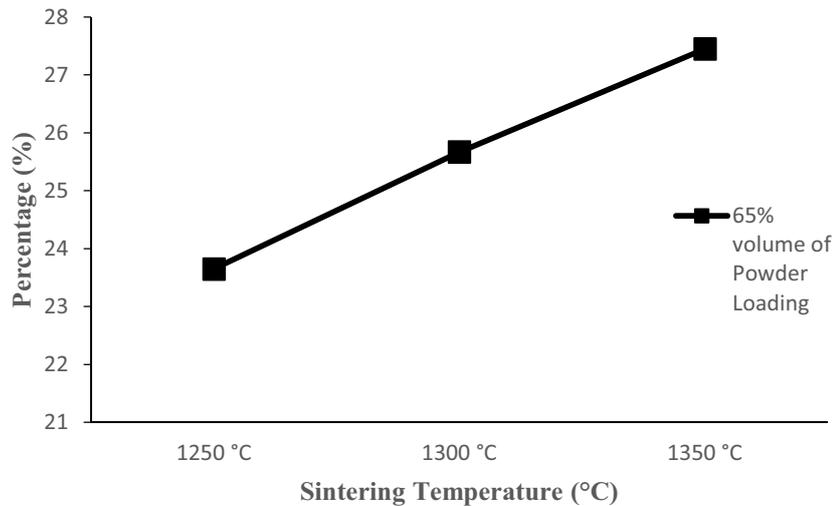


Figure 4. Shrinkage amount at different sintering temperature

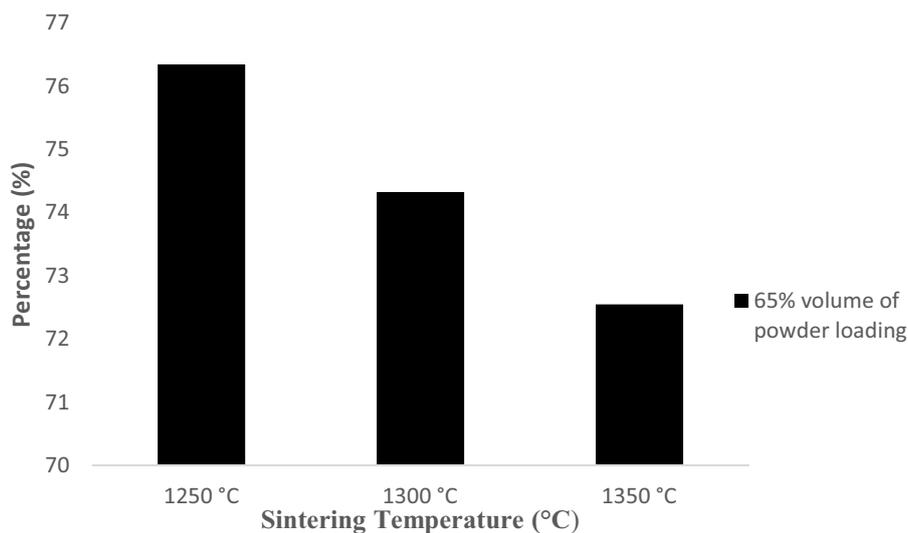


Figure 5: Percentage of dimensional accuracy for sintered compact at different sintering temperature

Figure 6 shows the hardness value of the sintered samples, showing sharp increase trend in hardness value from sintered temperature 1250 to 1350 °C. Sintered compact at sintering temperature 1250 °C recorded the lowest value of hardness, meanwhile compact at 1350 °C is the hardest one. By increasing the temperature, metal powder tends to melt and particle bonded together, after that penetrate porosity and fill large portion on it, helps to increase hardness [18]. Low level of porosity of CoCrMo (1350°C) sample can be assumed as the main factor of the high hardness value for that sample [19, 20].

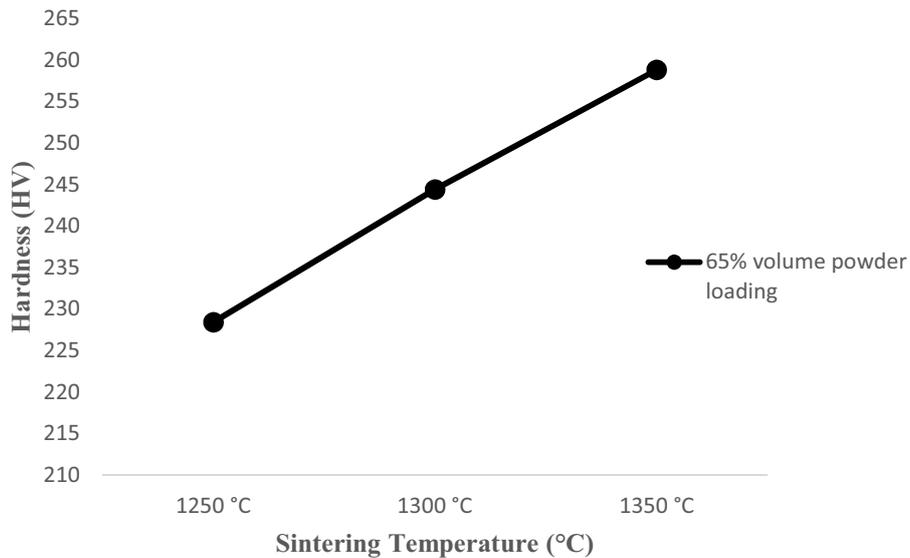


Figure 6. Hardness Value at different sintering temperature

4. Conclusions

From these three sintering temperatures tested, 1250 °C provided poor result in terms of microstructure of the sintered sample, leading to poor quality of mechanical properties as well, and it recorded lowest amount shrinkage. Sintering at 1350 °C recorded the highest hardness result showed that mechanical properties improved by the increasing the sintering temperature. Less amount of pores were detected on the sample sintered at 1350 °C, leading to good result for microstructure. But, it also led to the beginning loss of dimensional accuracy, where it recorded the highest amount of shrinkage. All sintering temperatures recorded below 80 % of dimensional accuracy where it does not meet the objectives of Powder Metallurgy; to produce near net shape components and below 5 % shrinkage that suggested by Heaney et al[15]. For next study, result will be improved by reduce sintering temperature lower than 1200 °C in order to achieve main goal in powder metallurgy method; to produce near net shape product. Sintering temperature was the parameter that strongly give impact on microstructure, physical and mechanical properties.

Acknowledgement

It is a great pleasure to acknowledge my deepest thanks and gratitude to my Supervisor and Post-graduate students at *Green Research for Advanced Materials (GRAMs) Laboratory, Human Engineering Focus Group, Faculty of Mechanical Engineering, Universiti Malaysia Pahang*, for their kind endless help and support in completing the project.

References

- [1] Y. Chen Y L, S. Kurosu, K. Yamanaka, N. Tang, A. Chiba 2014 Effect of Microstructures on the Sliding Behaviour of Hot-Pressed CoCrMo alloys *Wear* **319** 200-10
- [2] Z. Doni A C A, F. Toptan, F.R. Gomes, A. Ramalho, M. Buciumeanu, L. Palaghian, F.S. Silva, 2014 Dry sliding and tribocorrosion behaviour of hot pressed CoCrMo biomedical alloy as compared with the cast CoCrMo and Ti6Al4V alloys, *Mater. Des* **52** 47-57
- [3] Z. Doni A C A, F. Toptan, L.A. Rocha, M. Buciumeanu, L. Palaghian, F.S. Silva 2015 Tribocorrosion behaviour of hot pressed CoCrMo-HAP biocomposites *Tribology Int*
- [4] K. Yamanaka M M, A. Chiba 2014 Effects of nitrogen on microstructural evolution of biomedical Co-Cr-W alloys during hot deformation and subsequent cooling *Mater. Des* **57** 421-5

- [5] Koizumi Y, Suzuki, S., Yamanaka, K., Lee, B.S., Sato, K., Li, Y., Kurosu, S., Matsumoto, H., Chiba, A., 2013 Strain Induced Martensitic Transformation Near Twin Boundaries In A Biomedical Co-Cr-Mo Alloy with Negative Stacking Fault Energy *Acta Mater* **61** 1648-61
- [6] Muhammad Aslam F A, Puteri Sri Melor Binti Megat Yusoff, Khurram Altaf, Mohd Afian Omar, Randall M. German 2016 Powder Injection Moulding of Biocompatible Stainless Steel Biodevices *Powder Technology*
- [7] M. Javanbakht E S, M.J. Hadianfard 2016 The effect of sintering temperature on the structure and mechanical properties of medical-grade powder metallurgy stainless steels *Powder Technology* **Volume 289**
- [8] Hausnerová B 2011 Powder injection moulding- An alternative processing method for automotive items. *New Trends and Developments in Automotive System Engineering, Chiaberge M. (Ed.), InTech* pp.129-45
- [9] A. Arifin e a 2014 Material processing of hydroxyapatite and titanium alloy (HA/Ti) composite as implant materials using powder metallurgy: a review *Mater. Des* **55** 165-75
- [10] Jue Liu L C, Hairong Liu, Yongsheng Li , Hailin Yang, Jianming Ruan 2016 Microstructure, mechanical behavior and biocompatibility of powder metallurgy Nb-Ti-Ta alloys as biomedical material *Materials Science and Engineering C* **C17** 512-9
- [11] German R M 2008 PIM breaks the \$1 bn barrier *Metal Powder Report* **Vol.63** pp.8-10
- [12] Karimpoor A A, Erb, U., Aust, K. T., Palumbo, G. 2003 High Strength Nanocrystalline Cobalt with High Tensile Ductility *Scr. Mater* **49** 651-6
- [13] Edalati K, Toh, S., Arita, M., Watanabe, M., Horita, Z. 2013 High Pressure Torsion of Pure Cobalt: HCP-FCC phase Transformations and Twinning during Severe Plastic Deformation *Appl. Phys. Lett.* **102** 1-5
- [14] B.S. Becker J D B 1999 Cobalt-Base Alloys for Biomedical Applications *ASTM STP 1365* 47-61
- [15] Heany. D 2012 Handbook of Metal Injection Moulding
- [16] Boljanovic V 2010 In Metal Shaping Processes: Casting and Molding, Particulate Processing, Deformation Processes, Metal Removal *Powder metallurgy* pp. 75-106
- [17] Koizumi Y, Suzuki. S, Yamanaka. K, Lee. B.S. et al 2013 Strain Induced Martensitic Transformation Near Twin Boundaries in A Biomedical Co-Cr-Mo Alloy with Negative Stacking Fault Energy, *Acta Mater* **61** 1648-61
- [18] Wilson Correa Rodriguez L R B, Lirio Schaeffer, Gerhard Knorschild, Fidel Romel Mallqui Espinoza 2010 Powder Metallurgical Processing of Co-28% Cr-6% Mo for dental implants: Physical, Mechanical and Electrochemical Properties *Powder Technology* **Vol. 206** 233-8
- [19] Sh. Zangeneh H R L, A. Roshani 2012 Microstructure and Tribological Characteristics of Aged Co-28Cr-5Mo-0.3C alloy *Mater. Des.* **37** 292-303
- [20] M. Dourandish D G, A. Simchi, V. Firouzdar 2008 Sintering of biocompatible P/M Co-Cr-Mo alloy (F-75) for fabrication of porosity-graded composite structures *Materials Science and Engineering: A* **Volume 472** Pages 338-46