

ON THE EFFECT OF FEEDSTOCK PREPARATION ON THE CHARACTERISTICS OF WARM FORMED POWDER COMPACTS

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ABSTRACT

Powder compaction technology has advanced significantly over the past decades and is considered as an alternative lower-cost process to machining, casting, stamping, forging and other similar metal-working technologies. Feedstock preparation is considered a vital step in producing parts through the powder route because any inhomogeneity within the powder mass would cause inhomogeneous density distribution inside the green compacts, which in turn would cause fracture in the final products after sintering in the controlled environment. This paper presents the outcomes of an experimental investigation on the effects of feedstock preparation on the mechanical properties and microstructures of final products formed at above ambient temperature. A lab-scale uni-axial die compaction rig was designed and fabricated which enabled powder forming at elevated temperatures. Iron powder ASC 100.29 was mechanically mixed with different quantities of zinc stearate for 10, 30, and 60 minutes, respectively. Green compacts were generated by forming the prepared feedstock at 180°C through simultaneous upward and downward axial loading. The defect-free green compacts were subsequently sintered in an argon gas fired furnace and characterized for their physical and mechanical properties, and their microstructures were evaluated. The results revealed that the characteristics of the final products were affected by the feedstock preparation. The most suitable lubricant content and mixing time are found to be 0.4 wt% and 60 minutes, respectively. From this study, the proper zinc stearate content and mixing time were identified for the production of high quality products through the warm forming route. This study could be extended by using different types of base powder as well as lubricant.

Keywords: Feedstock preparation; warm powder compaction; sintering; product characterization.

INTRODUCTION

Nowadays, many engineering parts are produced by powder metallurgy (P/M), such as transmission and gearbox steel parts for automotive, cemented carbides and high speed steel parts, magnets and soft magnetic materials, fine ceramics, etc. (Baccino & Moret, 2000). P/M has advantages over other manufacturing processes mainly due to the elimination of secondary operations such as machining compared to wrought products (Chawla et al., 2011), and it promotes the combination of weight saving because of the low density of aluminum and material saving through near net-shape processing attributes (Mann et al., 2011; Kumar, & Singh, 2010). It is important to highlight that

dimensional stability as well as reliable mechanical properties make this process preferable to other manufacturing techniques. During powder forming, all the particles increasingly collide with each other. The powder particles also have to move along the die wall under the increasing applied pressure. Both phenomena during forming generate friction, and further generate heat and inhomogeneous particle distribution. This results in inhomogeneous density distribution inside the green compact that leads to internal crack generation during the ejection process. Therefore, to reduce this problem, an additive or lubricant has been an option to add inside the powder mass or/and adhere on the die wall. Furthermore, in preference to higher physical and thermal loading during the powder compaction process, lubrication is another option for increasing the density (Nor et al., 2008) and density uniformity of green compacts (Jiang et al., 2001). Two types of lubrication are usually practiced, i.e., admixed lubrication and die wall lubrication. The addition of lubricant admixed with powder mainly reduces inter-particle friction while lubrication in the interface of the powder with the die wall minimizes the die wall friction. The admixed lubrication method has been largely preferred by P/M industries due to its characteristic high production rate, cost effectiveness and applicability to all types of die. Admixed lubrication is effective in densification at relatively low compaction pressure (Simchi, 2003). The lubricant particles inside the powder mass are displaced together with the other metal particles when subjected to compaction pressure. The location and distribution of the lubricant play an important role in reducing the inter-particle friction. While a denser region is usually found at the powder–die wall interface, the capability of the lubricant to disperse out from the powder mass and reduce die wall friction is indispensable. Furthermore, the trapped lubricant during compaction has been known to vaporize during sintering, thus generating pores (Saha & Apelian, 2002), which greatly reduces the load-bearing area of a component. It is important for this admixed lubricant to be removed completely from the parts at the early stage of sintering (Babakhani, Haerian, & Ghambari, 2006) or during the compaction process if possible (Babakhani & Haerian, 2008).

The final stage of the powder metallurgy process is sintering. Conventional sintering or solid state sintering is the bonding of powder particles by molecular or atomic attraction in the solid state, by application of heat, causing strengthening of the powder mass and possibly resulting in densification and recrystallization by transport of materials (Hausner, 1979). This technique applies high heat treatment at a temperature below the melting point of the main powder constituent in the range of 60–80%, for a certain duration of time, in achieving the desired interaction among metal particles (German, 1996). During sintering of the powder compact, bonding among metal particles takes place as the main interaction among them. This process enhances the bond strength and provides a good arrangement of powder particles inside the compact. The metal powder compact undergoes three phases during the sintering process as follows: (i) neck growth proceeds rapidly but powder particles remain discrete, (ii) densification starts where the structure recrystallizes and powder particles diffuse into each other, and (iii) pores inside the powder compact become spheroid and densification happens at a much slower pace (Srinivasan, 2002; Vagnon et al., 2006).

The characteristics of the sintered product are affected by the different formulation of the lubricant added during feedstock preparation. A high weight percent of lubricant weakens the characteristics of the sintered product because it vaporizes and leaves pores during sintering, while a low weight percent of lubricant results in reduction of the flowability of the powder mass during compaction (Rahman & Nor,

2006; Rahman, Nor, & Rahman, 2011). Mixing time is also another parameter in feedstock preparation which plays an important role. A longer mixing time leads to the agglomeration of powder mass, whereas a shorter mixing time is not enough for the lubricant to fully assimilate with the metal powder to make a coherent powder mass that is ready to be shaped. Both situations cause the inhomogeneous density distribution inside the green compacts, which further causes the shape distortion of the final products. Thus, proper feedstock preparation is required for the production of high quality sintered components formed through the warm powder compaction route. So far, no study has been found in the literature on the proper feedstock preparation using iron as base powder for the production of near-net shape yet high quality mechanical components through the warm compaction route. Therefore, the objective of this paper is to investigate the effect of feedstock preparation in terms of lubricant content and mixing time on the characteristics of the sintered product formed through the warm compaction route.

MATERIALS AND METHOD

The main powder constituent used in the powder compaction experiment is iron powder ASC 100.29 which has a particle size range of 20–180 μm . Zinc stearate ($\text{C}_{36}\text{H}_{70}\text{O}_4\text{Zn}$) was used as lubricant due to its suitability (Ward & Billington, 1979). The compaction experiment was conducted using a custom-made T-15 compacting machine which uses the principle of hydraulic motion in order to transfer the load from the top and bottom punches. Four point heaters (50 Watt each) were attached to the cylindrical shape die. Zinc stearate was added to the iron powder in amounts of 0.2, 0.4, 0.6, 0.8 and 1.0 wt%. The powder mass was then mixed mechanically for three different durations of time i.e., 10, 30, and 60 minutes, respectively. The feedstock was then filled inside the cylindrical shape die and heated to 180°C for 30 minutes. The filled powder mass was then formed by applying a pressure of 380 MPa simultaneously with the top and bottom punches. The green products were then sintered in an argon gas fired furnace at 1000°C for 60 minutes at a rate of 10°C/minute. The sintered products were characterized in terms of dimensional stability, relative density, bending strength, and Rockwell hardness. The microstructures of the products were evaluated using scanning electron microscopy (SEM).

RESULTS AND DISCUSSION

Figure 1 shows the relative density of the as-compacted samples at different lubricant contents and mixing times. It is evident that increasing the amount of lubricant increased the relative density, while the mixing time had a different impact on density at different lubricant contents. Increase in zinc stearate provides less inter-particle contact among the iron powder particles in the powder mass, so more relocation and displacement of particles occurs, which leads to an increase in density. However, it has been observed that there is no increase in density when there is a further addition of 0.8 wt% zinc stearate, which is believed to be due to the particle inter-locking inside the powder mass. The mixing time proved to vary the consolidation process by means of providing the optimum positioning of zinc stearate inside the powder mass. The initial location of the zinc stearate determines the overall displacement and rearrangement of the iron particles.

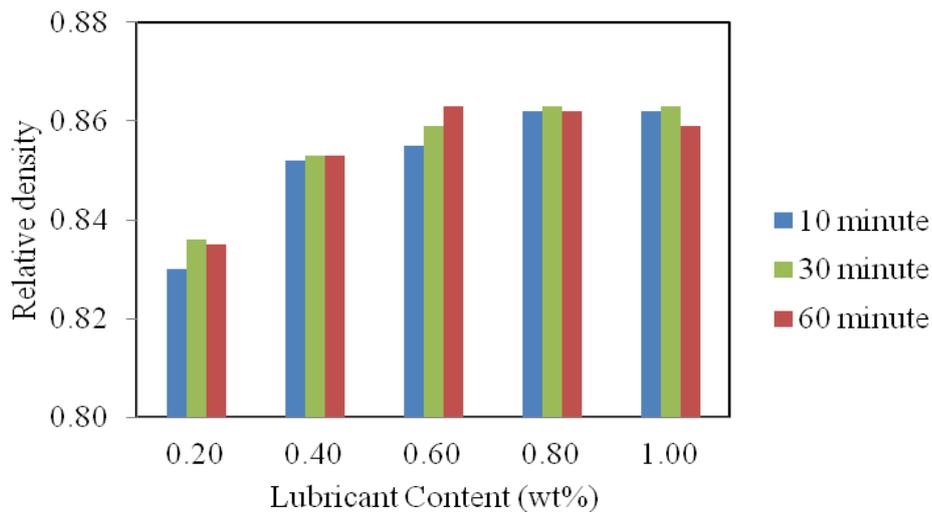


Figure 1. Effect of lubricant content and mixing time on the density of green compacts.

Figure 2 shows the sintered density at different lubricant contents and mixing times, while Figure 3 shows the green to sintered density changes. It can be observed that 0.2 and 0.4 wt% zinc stearate increased the density of the powder compacts during sintering, while at a lubricant content above 0.4 wt%, the sintered density reduced. More zinc stearate inside the powder compact leads to the vaporization and burning off of the zinc stearate during sintering, which leads to pore evolution by means of trapped gas, thus expanding the sample's dimensions, which is known as sample swelling. The effect of mixing time can reasonably be evaluated in the samples having 0.2 and 0.4 wt% lubricant, as the densification occurs at this range, where it is evident that 30 minutes mixing time increased the densification process. This is believed to be due to the flowing out process of zinc stearate from the powder mass during compaction.

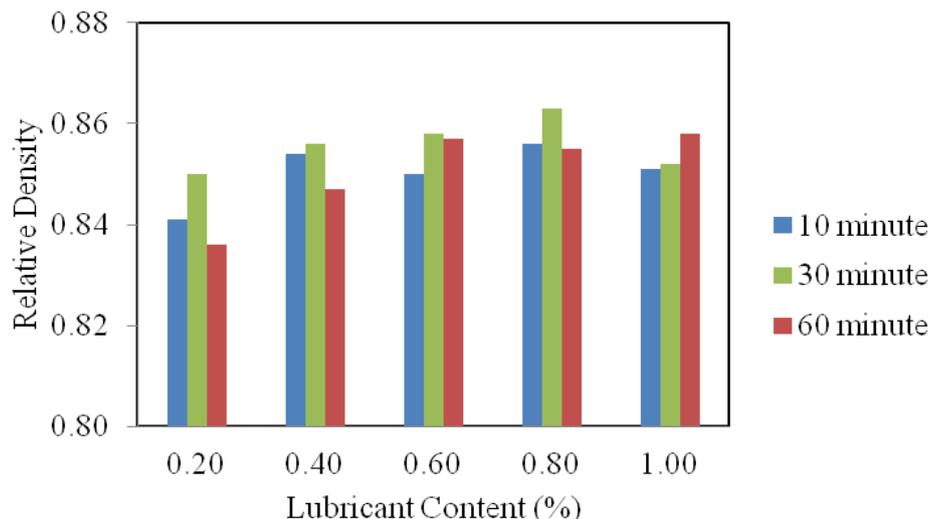


Figure 2. Effect of lubricant content and mixing time on the density of sintered products.

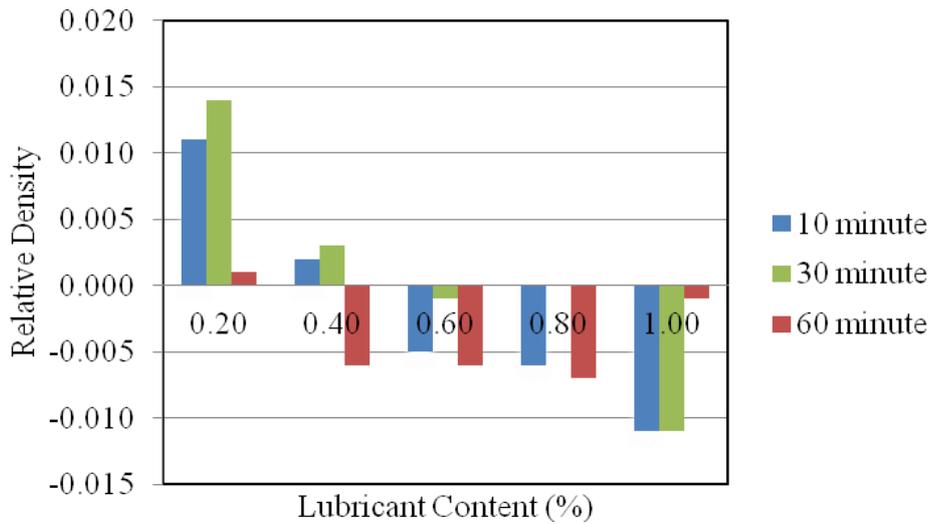


Figure 3. Changes of relative density from green to sintered products.

The bending strength of the sintered product is presented in Figure 4, where 0.4 wt% of zinc stearate is found to produce products having the highest strength compared to other products. Therefore, 0.4 wt% lubricant content can be considered as the optimum amount of lubricant for maximizing the displacement out of the lubricant during compaction. As bending strength implies the bonding among iron particles inside the product, it was proved that less zinc stearate was left inside the 0.4 wt% sample during sintering. Figure 5 shows the microstructures of the sintered products, where the feedstock was prepared by mixing 0.4% zinc stearate for 10, 30 and 60 minutes. For the 10 and 60 minutes samples, larger inter-connected pore are visible, which is proof of the existence of zinc stearate after the compaction process.

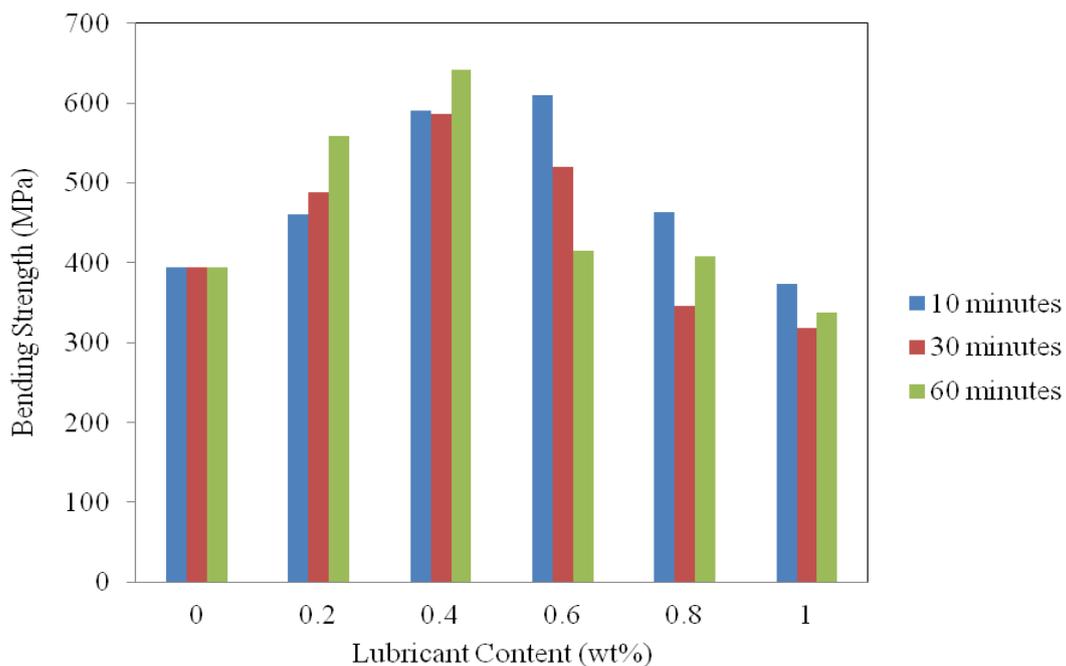


Figure 4. Bending strength of sintered products.

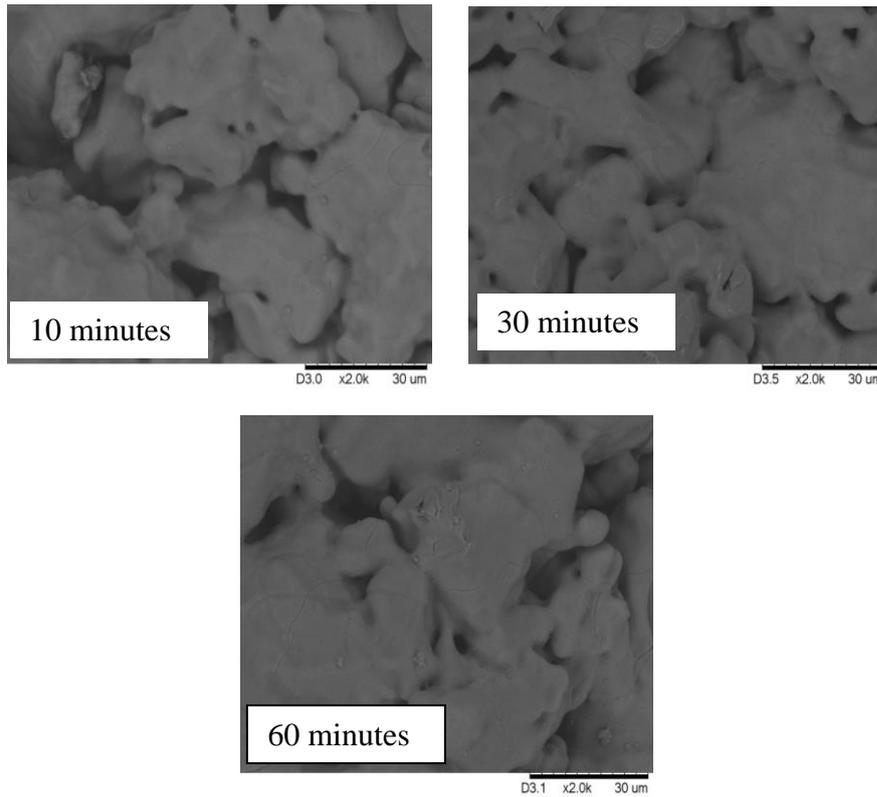


Figure 5. Microstructures of sintered products.

Figure 6 shows the hardness of the sintered product for all mixing times and lubricant contents. The highest value obtained is at 0.4% zinc stearate for 30 minutes mixing time, which is 108.9 HRF. The hardness of the sintered products only indicates the bonding of particles at the surface. Lower hardness means more pores are at the surface as a result of zinc stearate burning off during sintering, which leads to reducing the bonding among iron particles at the surface. Therefore, 30 minutes mixing time is shown to increase significantly the hardness of the products, which indirectly implies that this mixing time provides a good arrangement of lubricant particles during compaction.

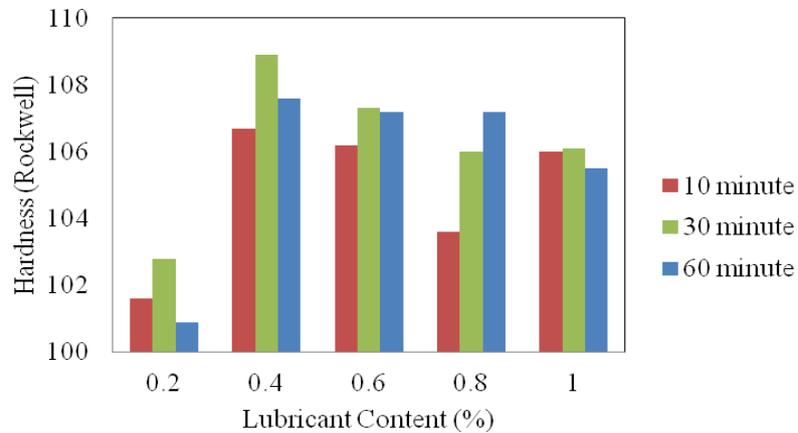


Figure 6. Hardness of sintered products.

The above results are further supported by the dimensional changes shown in Figure 7. In the case of 0.2–0.6 wt% zinc stearate content, sintered samples were found to shrink after sintering, while swelling occurred for the zinc stearate content of 0.8 and 1.0 wt%. The swelling is caused by the vaporization of zinc stearate during sintering because the gases produced from the zinc stearate are trapped inside the sample. As sintering proceeded, the pressure increased in the pore which led to pore expansion, and thus the samples swelled.

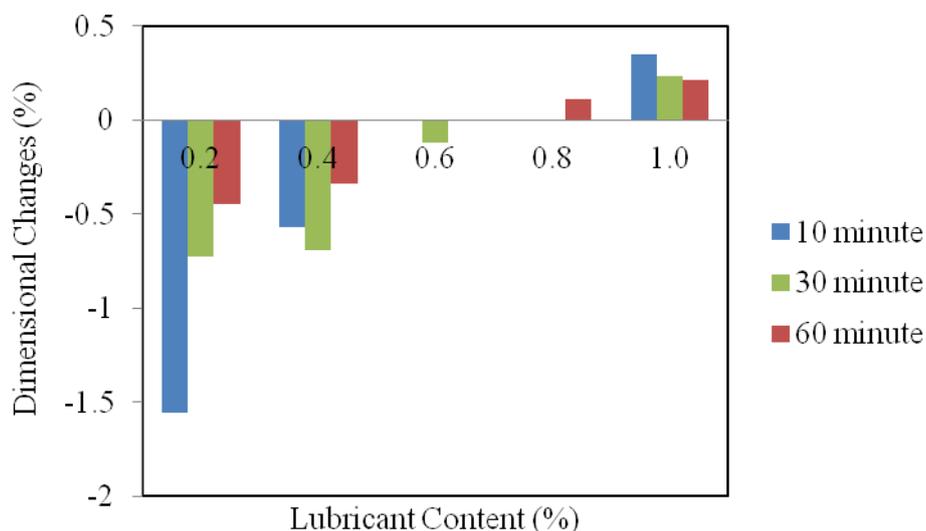


Figure 7. Dimensional changes of the sintered products.

CONCLUSIONS

This study found that the characteristics of sintered products are significantly affected by the feedstock preparation. The consolidated powder mass was markedly improved by increasing the amount of lubricant, while the mixing time decreased the lubricant burning off inside the powder compact. The strengthening of the bonding among particles inside the powder compact was also enhanced by mixing time. It was found that 0.4 wt% of zinc stearate mixed for 30 minutes with iron powder produced samples with improved mechanical properties.

ACKNOWLEDGMENT

The authors wish to thank the Ministry of Higher Education (MOHE), Malaysia for funding this research study under 08012011 ERGS.

REFERENCES

- Babakhani, A., Haerian, A., & Ghambari, M. (2006). On the combined effect of lubrication and compaction temperature on properties of iron-based P/M parts. *Materials Science and Engineering A*, 437, 360-365.
- Babakhani, A. & Haerian, A. (2008). Effect of carbon content and sintering temperature on mechanical properties of iron based PM parts produced by warm compaction. *Powder Metallurgy Progress*, 8(2), 156-163.
- Baccino, R. & Moret, F. (2000). Numerical modelling of powder metallurgy process. *Materials and Design*, 21, 359-364.

- Chawla, N., Murphy, T. F., Narasimhan, K. S., Coopman, M., & Chawla, K. K. (2001). Axial fatigue behavior of binder-treated versus diffusion alloyed powder metallurgy steels. *Materials Science and Engineering A*, 308(1-2), 180-188.
- German, R. M. (1996). *Sintering theory and practice*. New York: John Wiley.
- Hausner, H. H. (1979). Discussion on the definition of the term 'sintering'. In M. M. Ristic (Ed.), *Sintering - New developments*. Amsterdam: Elsevier.
- Jiang, G., Daehn, G. S., Lannutti, J. J., Fu, Y., & Wagoner, R. H. (2001). Effects of lubrication and aspect ratio on the consolidation of metal matrix composites under cyclic pressure. *Acta Materialia*, 49(8), 1471-1477.
- Kumar, S., & Singh, R. (2010). Investigation of surface properties in manganese powder mixed electrical discharge machining of OHNS and D2 die steels. *International Journal of Automotive and Mechanical Engineering*, 2, 181-199.
- Mann, R. E. D., Hexemer Jr., R. L., Donaldson, I. W., & Bishop, D. P. (2011). Hot deformation of an Al-Cu-Mg powder metallurgy alloy. *Materials Science and Engineering A*, 528(16-17), 5476-5483.
- Nor, S. S. M., Rahman, M. M., Tarlochan, F., Shahida, B., & Ariffin, A. K. (2008). The effect of lubrication in reducing net friction in warm powder compaction process. *Journal of Materials Processing Technology*, 207(1-3), 118-124.
- Rahman, M. M. & Nor, S. S. M. (2009). An experimental investigation of metal powder compaction at elevated temperature. *Mechanics of Materials*, 41, 553-560.
- Rahman, M. M., Nor, S. S. M., & Rahman, H. Y. (2011). Investigation on the effect of lubrication and forming parameters to the green compact generated from iron powder through warm forming route. *Materials and Design*, 32, 447-452.
- Saha, D. & Apelian, D. (2002). Control strategy for de-lubrication of P/M compacts. *International Journal of Powder Metallurgy*, 38(3), 71-79.
- Simchi, A. (2003). Effects of lubrication procedure on the consolidation, sintering and microstructural features of powder compacts. *Materials and Design*, 24, 585-594.
- Srinivasan, N. S. (2002). Dynamic study of changes in structure and morphology during the heating and sintering of iron powder. *Powder Technology*, 124, 40-44.
- Vagnon, A., Lame, O., Bouvard, D., Michiel, M. D., Bellet, D., & Kapelski, G. (2006). Deformation of steel powder compacts during sintering: Correlation between macroscopic measurement and in situ microtomography analysis. *Acta Materialia*, 54, 513-522.
- Ward, M. & Billington, J. C. (1979). Effect of zinc stearate on apparent density, mixing and compaction/ejection of iron powder compacts. *International Journal of Powder Metallurgy*, 4, 201-208.