

# AMM023 Micro Deep Drawing Process of Ti through the Medium of Iron Powder for Magnetic Hyperthermia Cancer Care

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# Abstract

Recently, a magnetic hyperthermia therapy one of the thermal cancer care techniques has attracted attention as a minimally invasive approach. It is well known that ferromagnetic implant materials in an alternating magnetic field causes heat generation. If the cancer tumor tissue around implants can be heated up to 43 deg. C, the cancer cells is killed because it corresponds to the statistical thermal death point. Since Fe have magnetic moment of  $2.2 \,\mu$ B, the large heat generation as implant miniature pellet is expected. On the other hand, development of cover layer is necessary because of unstable properties of Fe in the human body. This study demonstrates making of miniature Ti capsule which includes Fe powder inside as an implant for magnetic hyperthermia cancer care. Influence of particle size of Fe powder and friction to increase drawing ratio of Ti thin sheet have been discussed by experiment and numerical simulation by using distinct element method. Two types of loading pass for the test; Single stroke loading and Progressive loading have been proposed to fabricate  $\varphi 1.0$ mm Ti capsule. As the results, certain condition compatible with powder property is necessary to avoid rupture occurrence. More than 1 mm of height can be extruded in case of progressive loading with 32  $\mu$ m or lower particle size. The result of numerical simulation indicates that punch diameter become important because densification of powder layer depends on geometry of the tools. Finally, drawn length of 1.44mm has been obtained in optimal conditions which are determined by experiment and numerical simulation results.

Keywords: Micro forming, Deep drawing, Powder compaction, Distinct element method, Hyperthermia

#### 1. Introduction

Both the current status and the future forecast of the number of cancer patients are facing a severe situation. According to WHO reports there were 14.1 million new cancer cases, 8.2 million cancer deaths and 32.6 million people diagnosed with cancer (within 5 years of diagnosis) in 2012[1]. By 2030, 21.3 and 13.3 million people respectively will be affected by and die of cancer every year. In order to attain effective cancer therapy without side effects, new approaches which enable the patients to improve their quality of life have been explored for a long time. such However, major cancer therapies as chemotherapy, immunotherapy, radiation therapy and surgery still cause physical and psychological damage.

Recently, a magnetic hyperthermia therapy one of the thermal cancer care techniques has attracted attention as a minimally invasive approach [2-5]. It is well known that ferromagnetic implant materials in an alternating magnetic field causes heat generation. If the cancer tumor tissue around implants can be heated up to 43 deg. C, the cancer cells is killed because it corresponds to the statistical thermal death point [6]. The therapy is local and cell specific, so magnetic hyperthermia care takes great advantage as it can minimize the patient's physical and mental stress.

Magnetic hyperthermia method has two technical difficulties of implants. The one is the problem of its size. The implant pellet should be small to minimize damage in human body. The suitable shape is a needle type which have less than 0.8mm diameter for catheter

therapy. However, the needle with small mass of ferromagnetic element leads to insufficient heat generation. Therefore small needle or pellet with high calorific power is required for practical care. The other one is the unstable properties of implant materials. The implant materials should have high corrosion resistance in the human body because the magnetization of ferromagnetic materials is decreased by oxidation.

Some mechanical milling studies reports relationship between magnetic property and fine structure of ferromagnetic particles [7-10]. A study showed that coercivity which means width of magnetic hysteresis loop could be increased by both pulverizing and applying strong strain for particles. On the basis of the investigation, we tried to improve heat generation of Fe particles in alternating magnetic field by increasing coercivity using a planetary ball mill treatment. Fe have magnetic moment of 2.2 µB which is larger than Ni and Co, therefore the large heat generation as implant pellet is expected. On the other hand, development of cover layer is necessary for Fe pellet because of its unstable properties as implant materials in the human body. Thus the fabrication method of miniature pellet which is a capsule of Fe particles and its mass production process will be demanded to make implant pellet with certain calorific power in alternating magnetic field. We assumed that Ti thin sheet can be drawn into a  $\varphi$ 1.0mm die hole through the medium of Fe Powder and becomes outer shell of powder capsule. In previous work [11], we

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suggested indent-extrusion process of powder layer with Ti sheet. As the results, capsulation of Fe powder particle as extruded-drawn pellet has been successfully achieved. It has been indicated that certain thickness of the blank and lubrication condition is necessary to avoid rupture occurrence. More than 1 mm in length can be drawn without rupture occurrence in case of repetition. It has been corroborated that preventing of compaction pressure concentration is important to achieve higher extruded-drawn pellet length.

In this study, the influence of particle size of Fe powder and frictional conditions to increase drawing ratio of Ti thin sheet have been discussed by experiment and numerical simulation.

## 2. Experimental Procedure

# 2.1 Deep drawing process of Ti thin sheet through a medium of Fe powder

Fig. 1 shows principle of the micro indentextrusion process with Ti thin sheet as suggested capsulation process. In the test, Ti blank was placed on die, and was covered by Fe powder layer without pressurizing. Then upper punch was indented on the Fe powder layer, and the blank can be drawn into die cavity as pressurized powder particles was extruded into die cavity from behind of the drawn blank. Since the extruded-drawn pellet capsule should have long length to apply as an implant pellet for magnetic hyperthermia cancer care, we have attempted 2 types of loading pass for the test; Single stroke loading and Progressive loading.. It is expected that progressive loading has an advantage of which extrusion of powder particle into die cavity is promoted since powder particle layer can be rebuilt while the punch is withdrawn from the layer in each loading stage. After indentation, extruded-drawn pellet has been observed by microscope and its height was measured to discuss the feasibility of capsulation process.

The die-set was installed on universal testing machine (Shimadzu co, ltd. UH500kN, Japan). Indentation force and stroke were logged by data sampling system of the machine. Allowable load is limited at 3000N by compression strength of the punch. The diameter of die cavity is 1.0mm with 55 deg. taper which correspond with 1.5mm diameter inlet to promote extrusion of powder particles. Indentation speed was set to 1 mm/sec.

Ti thin sheet was selected as pure titanium sheet by JIS type 1 with annealing heat treatment in 600 deg. C, 1 hour. Diameter of the blank is 3.5mm as the aimed height of the drawn pellet is 3.0mm. Thickness of the blank is 0.1 mm.

## **2.2 Experimental Conditions**

We have investigated the influences of punch diameter size, lubrication conditions and Fe powder particle sizes. Firstly, we attempted the single stroke loading with  $\varphi$ 2.4mm,  $\varphi$ 2.0mm and  $\varphi$ 1.5mm diameter punches. Progressive loading was applied for  $\varphi$ 2.4mm. Fe powder of 32-75µm particle sizes with 1.0wt% zinc Harmonized Engineering Technologies

stearate addition was tested. Die and Ti sheets was lubricated by MoS<sub>2</sub> (molybdenum disulfide) grease.

Secondary, we attempted to change the lubrication conditions between Ti sheet and Die which are dry condition, MoS<sub>2</sub> grease, PVC (Polyvinyl chloride) sheet and complex of MoS<sub>2</sub> grease and PVC sheet. Thickness of PVC sheet is 2.0µm.

Moreover, we tried several conditions of lubrication between punch and Fe powder particles with  $\varphi$ 2.4mm punch in single stoke.

## 2.3 Application of Distinct Element Method

To discuss the mechanism of suggested process, numerical simulation has been demonstrated. Distinct element method code PFC2D (Itasca co, ltd. USA) was used. Fig. 2 shows example of a simulation model. Fe powder and Ti sheet is mentioned as green particles and blue particles. Note that Ti sheet is simulated as a collection of 32µm diameter particles which are bonded with certain strength.  $\Phi$ 1.5mm and 2.0mm punch diameter was tested in the simulation.



Fig. 1 Schematic view of micro deep drawing process through the medium of powder layer



process through the medium of powder layer

# 3. Results and Discussion

# 3.1 Influence of Punch Diameter

Fig. 3 shows variation of extruded-drawn length of the capsule in different punch diameter. Drawing

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length increases with increasing punch pressure in single stroke indentation. As compared with  $\varphi$ 1.5mm punch,  $\varphi 2.0$ mm and  $\varphi 2.4$ mm punches shows longer length (Fig. 3(a)). In case of progressive indentation with  $\varphi$ 2.4mm punch (Fig. 3(b)), the drawing length increases more than 300µm in the same punch pressure. It is considered that since Fe particles are supplied intermittently, particles extruded into die cavity promote drawing of Ti sheet. By the 6th indentation, drawing length exceeded 800µm. However, the rapture of Ti sheet arose in 7 times or more of the numbers of indentations. Since the rupture occurred in die shoulder, it is considered that consolidated particles fasten Ti sheet on die by increasing indentation number. Thus the deformation mode changed from bending to the stretch-expand forming with the processing. It is considered that φ1.5mm punch shows high pressure concentration as hold pressure on Ti sheet and drawing length decreases.



(Increment load pass: 245N–735N–1225N-1715N– 2205N and more for 7th-9th pass)

Fig. 3 Variation of extruded-drawn length in different punch diameter

# 3.2 Influence of Frictional Conditions

In the typical deep drawing process, friction coefficient in die and blank sheet should be minimized to increase drawing ratio. Therefore the complex lubrication of  $MoS_2$  grease and PVC sheet is expected to be most reliable. Fig. 4 shows extruded-drawn length of the capsule in different lubrication condition between die and Ti sheet. The result has agreed with the theory as the longer length has been obtained in complex lubrication.

Fig. 5 shows the influence of interparticle lubrication condition. It seems that there is no difference by the existence of 1.0wt.% zinc stearate addition for Fe powder layer for single indentation loading.



Fig. 4 Influence of lubrication condition in die and Ti sheet

(1.0wt.% zinc stearate addition for powder layer)



Fig. 5 Influence of lubrication condition in Fe particle layer

(MoS<sub>2</sub> grease lubrication for die and Ti sheet)

# 3.3 Results of Numerical Simulation

Fig. 6 shows results of simulation obtained by PFC2D. In the fig. 6(a), friction coefficient was set to 0.2 between die - sheet and particle - particle respectively. The result has nearly agreed with the real deformation aspect as a flange part loses touch with a die surface. In the 2nd indentation, thickness of sheet at die shoulder becomes thin. Fig. 6(b) shows the case of lower friction coefficient which set to 0.1 between die and sheet. Drawn length becomes about 10% longer than the case of 0.2, and reduction in thickness at die shoulder is restrained.

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Fig. 6(c) shows the case of larger particle size, 100 $\mu$ m diameter. Since larger particle size reads to lower drawing length, it seems that particle size should be minimized for this process.

In contrast with the general deep drawing theory, decrease in thickness of the drawn sheet at the tip of capsule did not occur in both of experiment and numerical simulation. This fact means that the deformation of blank sheet mostly occurs at die shoulder part. The case where additional hold pressure was applied on the flange part is shown in fig. 6(d). In this case, deformation mode seems like typical stretch-expand forming. Thus, it should not apply hold pressure actively in micro deep drawing process through the medium of powder layer.

With the consideration of experimental results mentioned in fig. 3(b), punch diameter become important because densification of powder layer depends on geometry of the tools. Fig. 7 shows the results on single loading indentation for the case of 2.0mm diameter punch. Since remarkable drawn height has been obtained, using a distinct element method will be reliable to determine the influence of tool geometry with certain lubrication condition. However the simulation results show quite larger drawn height than the experimental results. In future work, more precious material parameter setting with special consideration of constitutive model of Fe powder will be demanded to increase precision of analysis. Then hold pressure of blank and die shoulder angle should be optimized.

## 3.4 Experimental Result of Optimal Condition

Fig. 8 shows the longest drawing length obtained in optimal conditions determined by the experiment, which are;

- Particle size: under  $32 \mu m$
- Punch diameter:  $\phi 2.4mm$

Lubricant in die and sheet:  $MoS_2$  grease and PVC sheet

Lubricant in powder layer: Dry

Loading method: Progressive loading

Finally we have succeeded in obtaining 1.44mm drawn length. However, it is not enough for hyperthermia cancer care due to low heat generation ability. More optimization in accordance with clarify of the mechanism is required.

## 4. Conclusions

Micro deep drawing of pure Ti thin sheet through the medium of Fe powder layer has been carried out to make implant for hyperthermia cancer care. Influence of particle size of Fe powder and friction to increase drawing ratio of Ti thin sheet have been discussed by experiment and numerical simulation. The optimal conditions which are determined by experiment and numerical simulation results are qualitatively concluded as follows;

(1) The punch diameter which is twice the diameter of a die hole

(2) Fine particle size



Fig. 6 Result of numerical simulation by using distinct element method

(1.5mm punch diameter)



Fig. 7 Results on single stroke indentation for the case of 2.0mm punch diameter (Friction coefficient 2.0)



Length: 1.44mm

Fig. 8 Appearance of drawn capsule in optimized condition





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(3) Application of progressive indentation loading

(4) Low friction between die and blank sheet

Optimization of angle or radius in die shoulder, and punch diameter become important to control densification property of powder layer which will specify deformation mode of Ti sheet.

# 5. Acknowledgement

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