A compaction die has an essential role in the powder metallurgy process due to controlling and ease of processing. The shape and properties of die can significantly affect the features of the final part as the several steps and assessments are involved before design and fabrication of compaction die. This work is an effort to design and manufacture a compaction die that can successfully use for uni-axial compaction of NiTi powder. The design and development of die includes design consideration, 2D drawing, 3D model and processes involved in the fabrication of die. The die design was analyzed by ANSYS 19.1 software in a simulated environment, and the fabricated die was tested experimentally by preparing the composite sample successfully at 1000MPa compaction pressure using universal testing machine. Further, the compaction behaviour, density, compressive strength and hardness of developed NiTi composite have also evaluated.

Keywords: Powder metallurgy, compaction die, density, compressive strength, hardness

1. Introduction

Powder Metallurgy (PM) is a rapidly growing technique used for the development of most alloys and composites. It consists mainly three steps, as mixing of powdered materials, compress them into the desired shape, and then heating the compressed material in order to bind the material [1]. The PM process is also used to make specially designed material or part due to ease of processing and control, which are difficult to fabricate by other processes such as melting and forming [2, 3]. This process can prevent or significantly reduce the need for metal removal processes, thereby significantly reduce the manufacturing cost [4]. Another advantage of the PM process is that the components can be manufactured without decomposing or dissolving the material by avoiding the changes in material properties in the solid-liquid phase [5]. Therefore, powder processes are more flexible compare to other manufacturing processes such as casting, extrusion, and forging due to controlled characteristics of products prepared using this technique [6]. Several studies reported that the porous solids, aggregates and inter-metallic composites are efficiently manufactured by PM process with improved material characteristics [7]. There are several methods available for pressing in which uni-axial compaction is the simplest method of powder processing. In
uni-axial compaction the powder is compacted by compress the metal powder in a close die chamber by applying high pressures in an axial direction [8]. Generally, the pressure of 150 MPa to 1000 MPa is usually used for metal powder compaction [9]. Typically the die is held in the vertical direction with the punch. The powder is then pressed into the desired shape and then ejected from the die chamber, followed by the sintering process at a specific temperature under controlled environment [10]. The significant limitations of pressure compaction process are higher porosity and circumferential micro-cracks developed on the surface of green compact due to friction generated between die wall and composite particles. In PM processed components, the micro-cracks are primarily originated during the compaction process and broadened during sintering [11-13]. Hence, present research work aims to design and develop pressure compaction die to compact NiTi composite powder with minimum porosity and micro-cracks. The design of die has analyzed by ANSYS 19.1 software under simulated conditions. Further, the performance of the die has been examined experimentally based on compaction behaviour of NiTi composite powder, porosity induced during compaction and mechanical properties by applying maximum pressure through a universal testing machine.

2. Material and methods

2.1 Die material

High carbon (C) and high chromium (Cr) cold work steel, generally known as D3 die steel was used for fabrication of compaction die. The studies shows that the higher hardness and wear resistance of die steel make it suitable for tooling applications such as powder metallurgy tooling, draw dies, and blanking and forming tools[13]. The compositional details of D3 steel is given in table 1. Singh et al. [14] have reported that the density and ultimate compressive stress of D3 steel is equal to 7.7 g/cm³ and 2151 MPa. The hardness, tensile strength and yield strength of D3 steel is varies from 30 to 69 HRC, 990 to 2900 MPa and 705 to 2400 MPa respectively based on the condition of heat treatment [15].

Table 1: Chemical composition of D3 steel [13]

| Element | C     | Mn | Si | Cr  | Ni | W | V | P  | S  | Cu |
|---------|-------|----|----|-----|----|---|---|----|----|----|
| Content (%) | 2.1 | 0.6 | 0.6 | 12  | 0.3 | 1 | 1 | 0.03 | 0.03 | 0.25 |

2.2 Design calculation

The compaction die was designed to develop the green compaction samples of NiTi composite with sample diameter 20 mm and thickness 5 mm. The design of compaction die is as follows:

Yield strength, $\sigma_y = 1300$ MPa [15]

Chamber diameter, $D_i = 20$ mm

Outer diameter of die, $D_o = 90$ mm

Working load $F = 320$ KN

Cross section area $A = (\pi/4) d_i^2 = 314$ mm²
Stress developed in plunger due to applied load of 320 KN

\[ \sigma_1 = \frac{F}{A} = \frac{320000}{314} = 1019.1 \text{ MPa} \quad \text{[Hence } (\sigma_1 < \sigma_y) \text{ safe working condition]} \]

Stress developed in die due to load applied on plunger

\[ \sigma_2 = \sigma_1 \left[ \frac{(R_o)^2 + (R_i)^2}{(R_o)^2 - (15)^2} \right] = 1019.1 \left[ \frac{(45)^2 + (10)^2}{(45)^2 - (10)^2} \right] \]

\[ = 1124.98 \text{ MPa or } 1125 \text{ MPa} \quad \text{[Hence } (\sigma_2 < \sigma_y) \text{ safe working condition]} \]

2.3 Drawing of die

A 2D and 3D model of the die and punch set with the above mentioned design parameters was prepared using SOLIDWORKS modeling software. The 2D drawing of die chamber, upper and lower punch with dimensions is shown in figure 1(a). The 3D model of die assembly is shown in figure 1(b) and separate die parts are shown in figure 1(c). A suitable clearance of 0.5 mm has also given in the punch to reduce the surface contact between punch and wall of die cavity and thus minimizing friction and wear between the mating surfaces of punch and die chamber.

Figure 1: Schematic drawing with (a) 2D and (b and c) 3D model of compaction die
2.4 Fabrication of die parts

A D3 die steel stock of 95 mm diameter and 50 mm diameter was procured from P. R store, Agra, India and cut into the required size for die chamber, upper and lower punch with power hack saw. The pre-machining of die chamber, upper and lower punch was done by facing, turning, drilling and boring operation on CNC lathe machine. Heat treatment of die cylinder and punch set was done in muffle furnace after the pre-machining in three steps as stress relieving, hardening and tempering. Stress relieving was done at 650 °C for one hour and cooled slowly. For hardening, the die parts were directly placed in a furnace preheated to 1000 °C and soaked for 25-30 minutes and then quenched in still air to harden it. The parts were placed in the furnace for tempering and the temperature of furnace is increased slowly to the desired tempering temperature (200 °C) and cooled slowly in furnace. Finally, the surface finishing operation of die parts was done using cylindrical grinder. The finished die chamber, upper and lower punch is shown in the figure 2.

![Figure 2: Finished die parts (a) die chamber, upper and lower punch, and (b) assembly of die parts](image)

3. Experimental analysis

3.1 ANSYS analysis

The designed die, upper punch and lower punch were analyzed for stress distribution using ANSYS 19.1. The analysis was selected as structural with the element type as round steel. The die chamber, both punches and NiTi powder were explored as .igs file from SOLIDWORKS 2017-18 and imported to ANSYS 19.1. The material properties of D3 steel and NiTi powder are given in table 2. All parts of die are meshed with each other and it is assumed that the 20 gram of NiTi powder was poured in die chamber. The pressure of 1000 MPa was applied on the punch and it displaced in downward direction inside the die chamber. The stress distribution of various die parts and compacted powder are shown in figure 3(a) to (e). The analysis shows that the upper punch and die chamber induced minimum stress equal to 1.9751*10^7 Pa and the maximum stress (1.1101*10^10 Pa) was induced in lower punch and powder.
Table 2: Mechanical properties of D3 die steel and NiTi powder

| Material          | Density (g/cm³) | Young's Modulus (GPa) | Poisson's Ratio |
|-------------------|-----------------|-----------------------|-----------------|
| D3 steel          | 7.7             | 190                   | 0.27            |
| NiTi powder       | 6.75            | 75                    | 0.33            |

Figure 3: Stress distributions in (Pa) on (a) upper punch, (b) die chamber, (c) lower punch, (d) powder and (e) assembly

3.2 Powder compaction

The fabricated die was analyzed experimentally by compacting the NiTi composite powder using 400 KN universal testing machine in material testing lab at Rajkiya Engineering College, Mainpuri, India. After sufficient lubrication the 20 gm NiTi powder was poured in die cavity the powder was compressed by applying pressure of 1000 MPa at uniform rate. After the compaction, the compressed green compact was ejected from the die cavity with the help of ejector and green sample was sintered 1100 °C in muffle furnace for 4 hrs 30 min at the heating rate of 15 °C/min. The die arrangement in UTM and green sample developed after compaction and sintering are shown in figure 4.
Figure 4: Image of (a) die arrangement on universal testing machine during compaction process and NiTi composite sample after (b) compaction and (c) sintering

3.3 Density

The green density of the sample was examined after compaction as well as sintering and compared with the theoretical density of NiTi composite powder. Theoretical density of NiTi composite powder was calculated using mixing rule. In addition, the green density of developed sample was measured using Archimedes principle in distilled water. The mass of the sample was measured on the electronic balance. The sample was attached to a thread and fully immersed in distilled water. The weight of the sample in distilled water was measured, whereby the loss of weight of the sample when suspended in distilled water is equal to the mass of fluid displaced, from which its volume can be calculated. Further, the porosity of sintered samples was analyzed by following formula:

$$\text{Porosity} = \{1 - (\text{sample density}/\text{theoretical density})\} \times 100$$  \[1\]

3.4 Compressive strength and hardness

The compressive strength and hardness of sintered sample was also examined. Compression test has carried out using 400 KN universal testing machine at room temperature. The compressive strength NiTi sample is investigated by single experiment mode in which a cylindrical shape sample with 19.48 mm diameter and 12.8 mm thickness is used for the compression test. The set up for compression testing was shown in figure 5(a). To determine the hardness, the composite sample was tested using a Rockwell hardness testing machine with a diamond indenter. The Rockwell hardness of NiTi composite is measured at a load of 150 kg for 15 s dwell time. An average value of 5 measurements on the composite sample was determined. The set up for hardness testing is shown in figure 5(b).
4. Result and discussion

4.1 Compaction behavior

The compaction behavior of NiTi powder was directly influenced by compaction pressure. The compaction behavior of NiTi composite powder is shown in figure 6. It shows higher displacement of punch in the beginning of compaction due to the larger space available between the particles of composite powder but at the higher load displacement become constant at the end of compaction due to green strength and less space developed between composite particles. It is observed that the NiTi composite powder has successfully compressed to maximum density without any considerable die deformation.

Figure 6: Compaction behavior of NiTi powder with respect to displacement versus load
4.2 Densification behavior

The density of developed sample was measured after compaction and sintering. The density of NiTi composite sample after compaction and sintering compared with theoretical density of NiTi composite is shown in figure 7. The results show that the density achieved after the compaction of sample (4.89 g/cm³) is lower than the theoretical density (6.75 g/cm³) of NiTi powder. The density of sintered sample (5.02 g/cm³) was slightly increased due to the formation of necking between the NiTi particles and vaporization of moisture and lubricant. The porosity of sintered NiTi composite sample has calculated with the help of equation 1. By calculation, 27.56 % porosity was found during the compaction process.

![Figure 7: Density of NiTi powder and sample after compaction and sintering](image)

4.3 Compression strength and hardness

Compression testing was performed on computerized UTM of capacity 400 KN. The sample was placed in between two flat plates and maximum failure load was recorded. The hardness test was performed on Rockwell hardness tester with diamond indenter. The result of compression and hardness test was shown in table 3. The results show that the sample prepared by compaction process by applying 1000 MPa load has achieved compressive yield strength, ultimate compressive strength and Rockwell hardness equal to 37.737 MPa, 115 MPa and 67 HRC respectively. It is observed that the compressive yield strength and ultimate compressive strength of NiTi composite is lower side due to induced porosity during the compaction process. However, the hardness of developed NiTi composite is near about of theoretical Rockwell hardness of NiTi alloy (58-65 HRC) [16].

| NiTi composite | Yield stress (MPa) | Compressive strength (MPa) | Hardness (HRC) |
|----------------|--------------------|---------------------------|----------------|
|                | 37.737             | 115                       | 67             |

Table 3: Compressive Yield strength, ultimate compressive strength and hardness of NiTi composite sample
5. Conclusion

The compaction die was successfully designed and fabricated to develop NiTi composite by the application of 1000 MPa load on UTM. The ANSYS analysis shows the minimum stresses are distributed on die and upper punch and maximum stresses are distributed on powder and lower punch. The experimental analysis shows that the NiTi powder was successfully compressed to make green compact with 4.89 g/cm$^3$ sample density and 27.56% porosity. The sintering further improves the sample density due to formation of necking between the composite grains and vaporization of moisture and lubricant. The mechanical properties of sintered sample have optimum value compare with the theoretical values of compressive yield strength, ultimate compressive strength and hardness. Finally, it can conclude that the NiTi composite can successfully developed by applying uniform pressure upto 1000 MPa using cold compaction die.

Acknowledgement

The authors are grateful and acknowledged to Dr. A P J Abdul Kalam Technical University, Lucknow, India (Ref. No: AKTU/Dean-PGSR/2019/1570, Project Id: 286, Dated: 10/07/2019) for the financial and technical support under Collaborative Research and Innovation Program (TEQIP III) for this research work.

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