

# Characteristics of titanium parts produced by powder injection moulding

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**Abstract:** The production and characteristics of a part produced from titanium hydride ( $\text{TiH}_2$ ) powder using the powder injection moulding (PIM) method are described. Use of hydrogen as a temporary alloying element in titanium alloys is an attractive approach to enhance processability including sintering, compacting, etc., and also to improve final mechanical properties. In this paper, the mechanical properties are presented, based mainly on the uniaxial compression resistance of sintering parts. The PIM parts were formed using three different sintering temperatures to verify the influence of dehydrogenated processes on final mechanical properties.

**Keywords:** Keywords: powder injection moulding, compression test, titanium hydride

## 1. INTRODUCTION

Powder injection moulding (PIM) is a combination of plastic injection moulding and powder metal sintering. The ingenious combination of existing technologies provided a means of producing ceramic or metal parts with the shape complexity of injection moulded plastic and the mechanical properties of machined parts. The first medical application was in the early 1980s, when a few orthodontic appliances were moulded. The use of PIM components by the orthodontic industry eased many of the medical industries' concerns about biocompatibility and corrosion resistance of this manufacturing method [1]. This method can produce an intrinsic surface texture (porous and rough) that must provide rapid alloying and sufficient bone ingrowth.

It is widely acknowledged that, in order to achieve clinical success, implanted materials must form a stable interface with surrounding tissue as well as being compatible with the mechanical properties of natural tissue. Unfortunately, the high loads required of many orthopaedic implants restrict the

selection of potentially useful materials. Currently, titanium is employed in many implant designs in either pure or alloy form because of strength, comparatively low stiffness, light weight, and relative inertness [2].

In examples of porous-surfaced regions implants formed by sintering Ti and Ti alloy implants, interparticle, and particle-substrate bonding occur by solid state sintering. A major concern in sintering these reactive metals is that furnace atmospheres are sufficiently non-oxidizing and, in addition, that unacceptable hydrogen embrittlement does not occur [3].

Titanium and conventional titanium alloys have a high affinity for hydrogen, being capable of absorbing up to 60 at. % hydrogen at 600°C, and even higher contents can be alloyed with titanium at lower temperatures [4]. Fortunately, the reaction of hydrogen with titanium is reversible owing to a positive enthalpy of solution in titanium, allowing hydrogen to be removed easily by vacuum annealing. At sufficiently high hydrogen contents, room temperature embrittlement provides an economic method for production of titanium powder, with the hydrogen then being removed by vacuum annealing [5].

The sintering of injection moulded, blended elemental powder was found to occur more readily when it was hydrogenated. It was suggested that hydrogen may clear the powder surface, thereby

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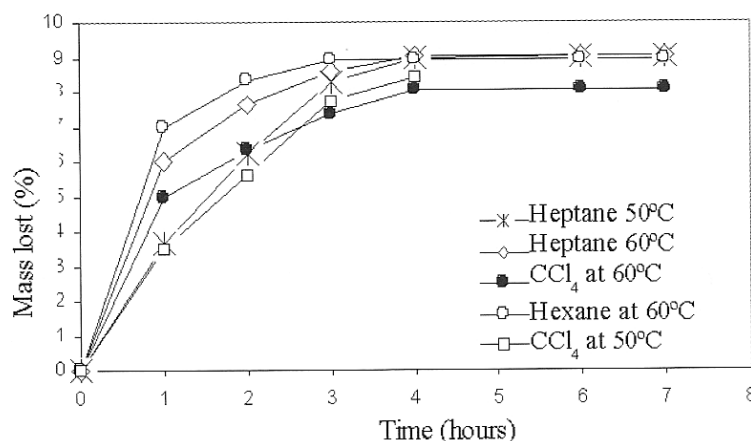


Fig. 1 Chemical extraction curves at different temperatures and organic solvents

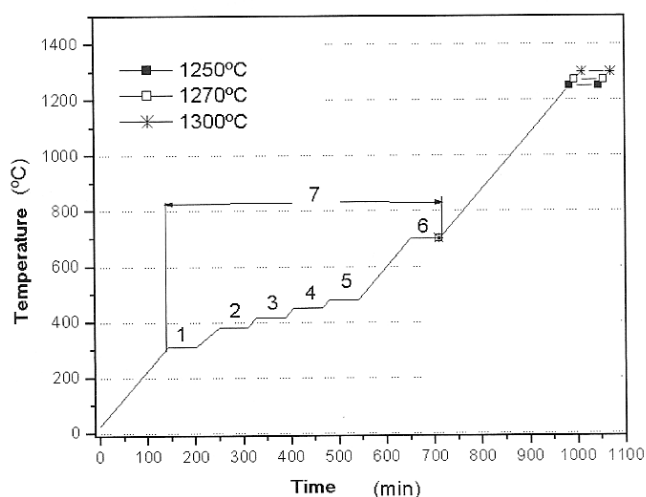


Fig. 2 Heating cycle used for dehydrogenation, thermal extraction, and sintering

promoting the better bonding of adjacent powder particles [5].

## 2. PRODUCTION OF SPECIMENS

In this work, one of many different forms is used to manufacture titanium parts. This technique consists of the mixture of hydride titanium powder ( $\text{TiH}_2$ ) with a binder system, the proportion of the mixture, in weight, being 20.9 %wt of binder system (polymeric materials – 11.27 %wt and waxes – 9.61 %wt) and 79.1 % of titanium hydride ( $\text{TiH}_2$ ) produced in the Metal-Forming Laboratory (UFRGS) by the hydrogenate-dehydrogenate (HDH) process [6]. The powder size of titanium hydride used was less than 45  $\mu\text{m}$ .

Mixing was performed in a double planetary mixer during a 1 h period at 190 °C. Subsequently, the mixture was injected using an injection moulding

machine model ARBURG® 220-S into a hot mould (70 °C). Near-net shaped cylindrical parts (diameter 4 mm) were moulded with 1600 bar injection pressure and 2000 bar post-pressure at 180 °C.

After moulding, the parts were chemically debound in different solvents and temperatures for comparison. Figure 1 shows the comparison between heptane and carbon tetrachloride at 50 °C and 60 °C, and hexane at 60 °C. The weight of parts was measured using an analytical balance with a 1 h frequency. It was observed that a total extraction of wax components occurs after 4 h in relation to the initial volume of the sample for all solvents and at all temperatures.

In a furnace with an argon 5.0 flux the second extraction step (thermal extraction for polymer compounds), dehydrogenation, and sintering were performed. The cycle is shown in Fig. 2. The heating rate used was 2 K/min. In thermal extraction the volumetric yield of gaseous hydrogen from the decomposition of  $\text{TiH}_2$  depends on the temperature and was performed on parts together with the decomposition of polymers of the binder system.

The hydrogenated powder can be consolidated with less energy than when non-hydrogenated powder is employed. This translates into a pressure reduction of 34 to 67 MPa or a temperature decrease of 110 to 140 °C. The sintering of an injection moulded piece using blended elemental powder promotes the better bonding of adjacent powder particles [7]. The volume of the piece shrinks because of the densification of parts during sintering.

The numbers presented (1, 2, 3, 4, 5, and 6) up to plateaus in Fig. 2 represent the temperatures that occur during the debinding of polymeric compounds of the binder system; number 7 represents dehydrogenation. Dehydrogenation occurs at the same time as debinding. The ideal time at distinct temperatures is still under study. After this

period, the parts were pre-sintered at 900 °C, sintered at three different temperatures (1250, 1270, and 1300 °C), and finally cooled down to the finished process.

After sintering in the furnace with an inert atmosphere, the weight percentage of the chemical composition of contaminants was analysed in a CS-444 for carbon, in a TC-436 for nitrogen and oxygen, and in an RH-402 for hydrogen (LECO® instruments). The results are shown in Table 1. The CS-444 uses infrared adsorption principle to determine the carbon composition and TC-436 and the RH-402 series utilize the inert gas fusion principle to determine the other elements presented.

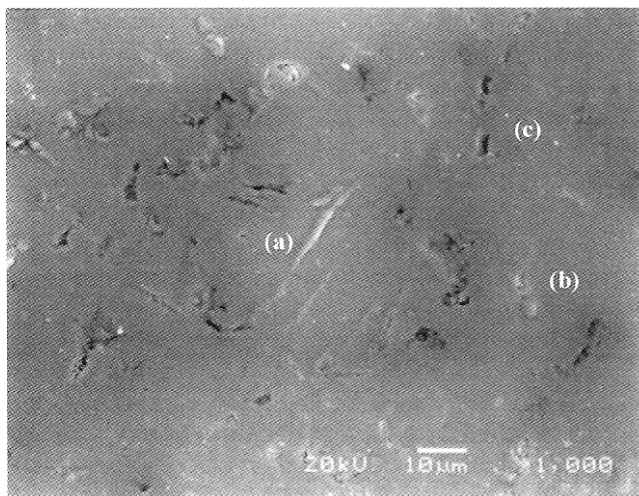
In the morphology of the sample shown in Fig. 3, it is possible to see evidence of a needle-shaped hydride phase and spherical regions of titanium carbide. This can be explained by the high residual values of carbon and hydrogen shown in Table 1. The relative density of sample was estimated as approximately 98 per cent of bulk titanium, using Archimedes' method.

### 3. COMPRESSION TESTS

The stress-strain test was performed using three groups of specimens, each consisting of 12 specimens. Three levels of sintering temperatures were used and compared with machined samples. Typical

**Table 1** Chemical composition of contaminants after sintering (%wt)

C	O	N	H
1.560	1.650	0.013	0.195

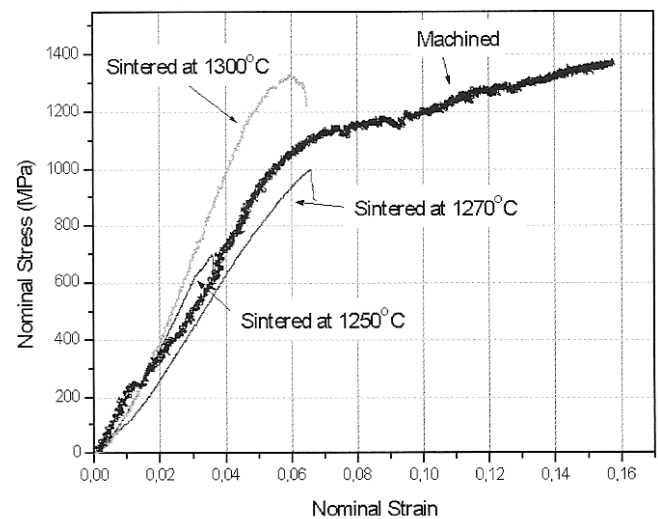


**Fig. 3** Electromicrography shown the morphology of the sample sintered at 1300 °C; (a) hydride phase, (b) spherical regions, and (c) intrinsic porosity

stress-strain curves for type machined (bulk) and the type sintered at 1250, 1270, and 1300 °C at strain rates of 0.06 mm/min are shown in Fig. 4. The compression test at room temperature (27 °C) was performed in the same direction as the principal direction of injection in this study. The curves show an elastic region at first, followed by a plastic region in the type machined. However, In types sintered at 1250, 1270, and 1300 °C the plateau does not occur. After the elastic region, flow stress rapidly decreased because the specimens were sintered. The results presented in Fig. 4 indicate a brittle behaviour of sintered samples owing to cited morphologic factors and production process (sintering temperature).

The relationship between sintering temperature and stress in the sintered specimens subjected to compression is presented in Table 2. The statistic results show a significant difference in mechanical response between 1300 °C and the other two sintering temperatures, but this does not appear at a different nominal strain interval between 1270 and 1300 °C.

The results of sintered samples at 1250 °C have lower compression strengths than the results of sintered at 1270 °C, 1300 °C and machined (max



**Fig. 4** Typical compressive stress-strain curve of the sintered titanium parts compared with machined titanium parts

**Table 2** Statistic results of ultimate compressive strength (UCS) in compression tests in accordance with the sintering temperature

Sintering temperature	1250 °C	1270 °C	1300 °C
Max UCS (MPa)	698	998	1305
Mean UCS (MPa)	242	304	512
Sd Er±	161	273	367
Number of experiments	13	13	9

UCS 1370 MPa). The comparison between ultimate compressive strength results of 1250 and 1270 °C sintering temperatures shows a non-significant difference.

Table 2 gives the mean value and the maximum value of compressive strength determined for each sintering temperature type, as obtained from at least nine experiments by stress-strain curves similar to those exhibited in Fig. 3. It can be observed that the results of both the maximum value obtained as well as the mean value determined for this mechanical property for the sintering temperature of 1300 °C are by far the highest values as compared with the results of the other two sintering temperatures.

#### 4. CONCLUSIONS

The reduction of strength owing to sintering temperature is associated with the porosity created in the process. In morphological terms, the needle-shaped hydride phase and the evidence of spherical regions of titanium carbide appear owing to components of the production process of feedstock. Also, as expected, the sintered parts present a brittle behaviour. The statistic of stress-strain compression tests indicates better results to the 1300 °C sintered samples in comparison with 1250 °C and 1270 °C and it probably means differences in the residual porosity

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