The investigation of die-pressing and sintering behavior of ITP CP-Ti and Ti-6Al-4V powders ☆

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ABSTRACT

This paper investigated the die-pressing and sintering behavior of ITP CP-Ti and Ti-6Al-4V powders produced by the Armstrong Process®. The ITP powders have an irregular coral like, dendritic morphology, with a dendrite size of approximately 2–5 μm. As-received as well as milled powders were uniaxially pressed at designated pressures up to 690 MPa to form disk samples with different aspect ratios. In the studied pressure range, an empirical powder compaction equation was applied to linearize the green density – pressure relationship, and powder compaction parameters were obtained. The ITP Ti-6Al-4V powder exhibited a significantly higher sinterability than the CP-Ti powder. This was explained to be due to the higher diffusivity of V in β-Ti at the sintering temperature. The Ti-6Al-4V samples with a green density of 71.0% increased to 99.6% after sintering at 1300 °C for 1 h. An ex-situ technique was used to track the powder morphology change before and after sintering.

1. Introduction

Powder metallurgy (PM) processes such as press-and-sinter, injection molding, and hot isostatic pressing are viable methods for producing near-net-shape components that require minimal machining, thus reducing manufacturing costs and increasing the material yield, which is commonly referred to as the "buy-to-fly ratio" in the aerospace sector [1–3]. PM methods have been used to make steel- and copper-based alloy parts for a wide range of applications [1]. However, in the case of titanium (Ti), the high cost of good quality powder (e.g. gas-atomized powder) has been a major stumbling block and has limited the commercial viability of Ti PM processing [4–7].

The Armstrong Process®, developed by Cristal US Inc./International Titanium Powder, Inc. (Woodridge, IL), produces high purity Ti and Ti-alloy powders in a one-step, continuous process [8–10] (The powder produced by this process will be referred to as ITP powder hence force). As Fig. 1 shows, metal chlorides are mixed and injected into a flowing stream of liquid sodium (Na) for reduction. The reaction by-product, NaCl, can be washed away by water. The Armstrong Process® has the potential to make Ti and Ti-alloy powders affordable, and has stimulated research at Oak Ridge National Laboratory (ORNL) to better understand Ti powder consolidation. Preliminary research has been performed on evaluating fabrication processes for near-net-shape forming of components using ITP CP-Ti and Ti-6Al-4V (Ti-64) powders. These processes include: die-pressing, cold isostatic pressing (CIP), sintering, hot isostatic pressing (HIP), and pneumatic isostatic forging (PIF) [11–13]. Fig. 2 shows a comparison of the buy-to-fly ratio for an aircraft component fabricated by conventional machining of a wrought Ti alloy block vs. a press-and-sinter PM method using ITP Ti-64 powder. In this example, the buy-to-fly ratio was reduced from 33:1 for standard processing to 4:1 for the PM approach. Powder produced by the Armstrong process®, used in combination with existing and developing PM technologies, has the potential to reduce the cost of finished Ti components by up to 50% [9].

This paper focuses on understanding the press-and-sinter behavior of ITP CP-Ti and Ti-64 powders. The relationship between green density and die pressure was evaluated and the sintering behavior of the two powders was compared.
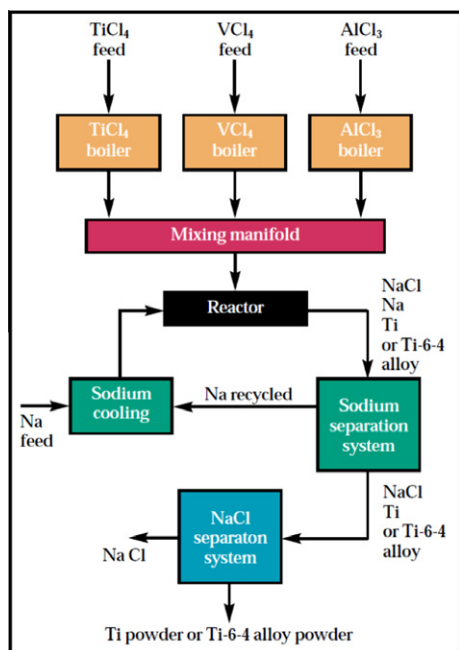
Fig. 1. Flow diagram of the Armstrong process for producing ITP Ti-6Al-4V powder [6].
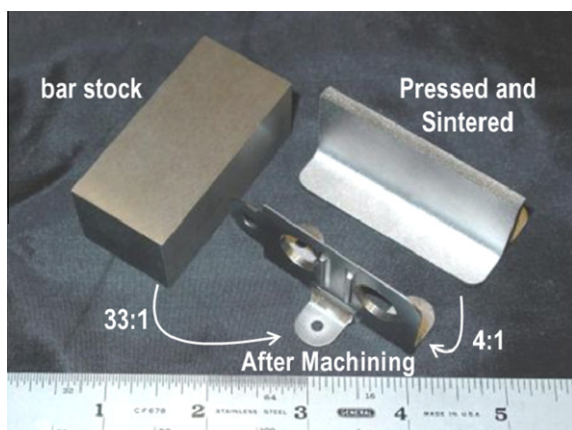

Fig. 2. The buy-to-fly ratio comparison between an aircraft component fabricated by conventional method (33:1) and PM method using ITP Ti-64 powder (4:1).

2. Materials and methods

The materials used in this study were CP-Ti and Ti-64 powders produced by the Armstrong Process® (Fig. 1a).The powders were used either in the as-received condition or after milling. For the milling operation, powders were ball-milled in ethanol for 1 h. using a polyethylene jar containing ~13 mm diameter yttria stabilized zirconia (ZrO_2) media. After the milled powder was dried, it was screened to minus 100 mesh (<140 μm), before being used for the current study. The powder chemistry was analyzed using inert gas fusion for oxygen (ASTM E 1409-08) and combustion infrared detection for carbon (ASTM E 1941-04). The morphology of the ITP powders was characterized using a JSM-6500F (JEOL, Inc.) scanning electron microscope (SEM). The specific surface area of the ITP powders was measured using the BET method with nitrogen gas as the adsorbate.

A cylindrical steel die with an inner diameter of 1.27 cm (0.5 in) was used for powder pressing, which was done at room temperature. The CP-Ti and Ti-64 powders were uniaxially pressed (double-action) over a range of pressures: 17, 34, 69, 172, 345, and 690 MPa (2.5, 5, 10, 25, 50, and 100 ksi). For the powders in the as-received condition, 2 g of powder was used for each sample. The milled powders were used to study the effect of the aspect ratio of pressed pellets on the green density, so the amount of powder used varied from 2.5 to 10 g depending on the specific aspect ratio. Cylindrical samples with nominal aspect ratios (thickness to diameter) of 0.5, 1, and 2 were obtained at each pressure. The density of the as-pressed samples was determined by measuring the mass with a balance and calculating the volume after measuring the dimensions with calipers.

The as-pressed samples were then sintered in vacuum (<5 × 10^{-5} Pa) at 1300 °C for 1 hour with heating and cooling rates of 10 °C/min. The density of the sintered samples was determined by means of Archimedes' principle (buoyancy method) using ethanol. An *ex-situ* SEM technique was used to examine the powder morphology before and after sintering. Selected as-pressed samples were broken to expose a fracture surface and a specific area on the surface was imaged using a HITACHI-S3400 (HITACHI, Inc.) SEM. The samples were then sintered and the same area was located to observe the powder morphology change after sintering.

3. Results and discussion

3.1. Powder characterization

Secondary electron-scanning electron microscopy (SE-SEM) photomicrographs of the as-received ITP CP-Ti and Ti-64 powders are shown in Fig. 3. The morphology of the two powders is similar. Both have an irregular coral-like, dendritic morphology, with a dendrite (primary particle) size of approximately 2–5 μm. The specific surface area for the as-received ITP CP-Ti and Ti-64 powders is 0.18 and 0.36 m²/g, respectively. Because of the irregular, agglomerated structure of the powders, the tap density of the as-received powder is 6–8%. The particle size distribution of the powders after ball milling is shown in Fig. 4. The milling process breaks up the agglomerates and significantly reduced the powder size, which increases the tap density to 30–32% [11]. The specific surface area also increased to 0.36 and 0.5 m²/g for the CP-Ti and Ti-64 powders, respectively. The chemical composition of the as-received powders is listed in Table 1.

3.2. Powder compaction and density-pressure relationships

Fischmeister and Arzt [14] suggested a three-stage model for the densification of spherical powders during die pressing:

Stage I: particle rearrangement, which occurs at the very beginning of compaction and does not contribute significantly to the densification of spherical powders.

Stage II: particle plastic deformation, in which particles are flattened and brought closer to form additional contacts.

Stage III: particle impingment, in which neighboring contacts on one particle impinge, making an increasing fraction of the volume harder to compress.

Poquillon et al. [15] studied the cold compaction behavior of a spongy iron powder which is quite similar in morphology to the ITP Ti-64 powder. Three deformation stages were clearly observed for the iron powder, with particle interlocking being the dominant factor, which increased with compaction pressure. In a previous study, the current authors used an *in-situ* technique to observe the deformation of CP-Ti powder [16]. A powder compression test was performed using a screw-driven tensile stage placed inside an SEM. The observed compression process consists of three stages: 1. Powder cluster rotation; 2. Powder cluster broken down to fill in large spacing; 3. Powders being squeezed to fill in the inter-particle spacing. The corresponding stresses for the first two stages were below 10 MPa. In the final stage of compression, the powders were deformed and individual particles were brought closer to each other. The lowest pressure in the current study was 17 MPa (2.5 ksi), hence the compaction behavior described in this study corresponds to stage III.

Density-pressure curves have been widely used to study the compaction behavior of powders [1,17]. There are four widely used empirical compaction equations which have been developed by Heckel [17], Kawakita [18], Ge [19], and Panelli [20]. A critical review of these equations [21] showed that the Panelli equation best represented the density-pressure relationship for the 22 different