



# Modelling compaction behaviour of nickel–phosphorus and nickel–boron electroless coated titanium powders



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## ABSTRACT

Electroless plating is used as an alternative approach to alloying the commercially pure titanium powders. Two types of irregularly shaped powders, which differ in particle size distribution, are coated with nickel–phosphorus (Ni–P) and nickel–boron (Ni–B). A novel method, combining aspects of the Kawakita and Alderborn approaches, has been employed in order to understand compaction behaviour. The overall consolidation is described by a Kawakita–Ludde relationship, and the transition pressures which demarcate the limits of rearrangement only and plastic deformation only are identified using a modification of the Alderborn relationship. A significant increase in green strength is achieved with the Ni–B coated powders, and this is attributed to a modification of friction conditions and the number of possible contacts, which lead to an increased contribution from plastic deformation. The Mohr–Coulomb model and Ohyanne equations are used to describe the contribution of the number of contacts on green strength.

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## 1. Introduction

Powder metallurgical (PM) processes such as uni-axial pressing and sintering, and powder rolling provide near net shape consolidation with close dimensional tolerance and a good surface finish. In addition, for titanium components, these routes eliminate the disadvantages of melting and casting, reducing the cost of further processing. PM alloys are produced either by a blended elemental approach or by using a pre-alloyed powder. As an alternative to the conventional powder blending process to introduce alloying elements, an electroless plating route can be used to coat the desired element onto the surface of the base powder. The major advantage of this process is that the alloying elements in the coating are intimately in contact with the titanium particle surface, and so, may diffuse more readily into the titanium matrix when sintered at elevated temperatures; this reduces the sintering times needed to achieve both full densification and a homogenous alloy microstructure [1]. In this paper the alloying elements investigated are nickel, phosphorus and boron. Nickel, a beta stabilizer, is rarely added to titanium, other than for its shape memory effect, due to its higher cost. However, nickel reduces the solidus temperature and is also a fast diffuser in titanium [2], which should accelerate sintering [3]. Boron in titanium results in microstructure refinement [4] leading to an increase in tensile strength and stiffness. The presence of phosphorus

in the titanium matrix imparts an improved corrosion resistance, and the phosphates formed on the surface during heating processes improve the biocompatibility of titanium [5,6].

The various theories of electroless plating are described elsewhere [7–11]. The factors that play a vital role in the deposition of a uniform metallic layer of an element on the powders include the working temperature, the pH of the solution and the concentration of the chemicals added to the bath. During the plating of nickel–phosphorus (Ni–P) and nickel–boron (Ni–B) layers onto titanium powders, the titanium particles are constantly agitated in aggressive acidic (for Ni–P) and alkaline (for Ni–B) solutions. This leads to additional surface modification, especially in rounding of asperities and overall surface smoothness.

The densification of powders during uni-axial compaction can be influenced by: (1) the morphology, size, and distribution of particles, (2) the hardness, and yield strength of the particles and their response to plastic deformation and (3) the stress distribution that governs the movement of the particle and the interlocking during compaction [12,13].

In a classical approach, density–pressure relationships are used to understand the compaction behaviour of powders. The first mathematical compaction model was developed by Walker [14,15] based on certain theoretical assumptions. A linear relationship between the logarithm of axial pressure,  $P_a$ , (units of  $P_a$ :  $\text{kg}/\text{cm}^2$ ) and volume ratio,  $V$ , of the compact was derived:

$$V = C - K \log P_a \quad (1)$$

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Implicit in the application of a linear relationship is the assumption that a single compaction mechanism is being observed, and so this simple approach cannot adequately represent compaction where multiple mechanisms are operative.

Subsequently, a number of similar compaction equations were proposed, which attempted to theoretically justify the possible mechanisms during powder compaction processing [16]. Many of these again tend to follow a linear profile to fit the compaction data, but an improvement is found when the widely used Kawakita–Ludde equation is applied [17]

$$\frac{1}{C} = \frac{D}{D - D_{app}} = \left(\frac{1}{ab}\right) \frac{1}{P} + \frac{1}{a} \quad (2)$$

where  $C$  is the relative increase in the density,  $a$  and  $b$  are constants and  $P$  is the compaction pressure,  $D$  is the relative density of the compact and  $D_{app}$  is the fill or the apparent density of the powder. The chief advantage of this model is that it predicts behaviour at lower pressures and high porosities accurately [18,19].

Alderborn [20–23] proposed a novel method to understand the compaction behaviour of powders and related compaction to green strength. It was suggested that the effective deformability of the particles plays a vital role during compaction and that the stages where the particles fragment and subsequently deform directly affect the final strength of the compact. The effective deformability of a compact is given by:

$$\frac{\sigma_t}{\sigma_{t,\varepsilon=0}} = \frac{1}{C_1} (P_{app} - P_{C1}) \quad (3)$$

where  $\sigma_t$  is the tensile strength of the compact,  $\sigma_{t,\varepsilon=0}$  is the strength at zero porosity,  $C_1$  is the effective deformability of the compact,  $P_{C1}$  is the pressure required to form a coherent tablet and  $P_{app}$  is the applied pressure. The term  $(P_{app} - P_{C1})$  is the effective compaction pressure that results in deformation of the particles.

In order to calculate the number of contacts that spherical particles make with the surrounding particles, Ohyanne [24] proposed the following equation:

$$q = \frac{2n(1-\varepsilon_t)}{\pi(1-\varepsilon)} \left\{ 200 \left( \frac{\mu\pi}{16} + \frac{1}{3} \right) \varepsilon^2 + 140 \left( \frac{\mu\pi}{8} + \frac{1}{2} \right) \varepsilon \right\} \quad (4)$$

where  $q$  is the radial stress on the compact,  $n$  is the effective number of contacts per 1/8th of the sphere,  $\mu$  is the global co-efficient of friction,  $\varepsilon$  is the relative change in density with respect to the apparent density, and  $\varepsilon_t$  is the relative change in density with respect to the maximum density (density at  $P_{max}$ ). The relative change in density values,  $\varepsilon$ , is calculated as follows:

$$\varepsilon = \frac{D - D_{app}}{D} \quad (5)$$

where  $D$  is the relative density of a compact at a compaction pressure and  $D_{app}$  is the apparent density of a powder. The value of  $\varepsilon_t$  is thus calculated as:

$$\varepsilon_t = \frac{D_{P_{max}} - D_{app}}{D_{P_{max}}} \quad (6)$$

where  $D_{P_{max}}$  is the maximum compaction density achievable, and requires a pressure  $P_{max}$ . The radial stress on the compact is calculated using the radial to axial stress ratio of 0.55 [25], where the axial stress is the compaction pressure.

The Mohr–Coulomb line has been widely used to describe the compaction behaviour of pharmaceutical tablets [26–28]. The studies relate the influence of cohesive force ( $d$ ) and the angle of friction ( $\beta$ ) between the particles in the compact to densification. The assessment comes

from the plot between hydrostatic stress ( $p$ ) and Von-Mises stress ( $q$ ), yielding a straight line up to the yield point of the compact, and relies on two simple green strength measurements, namely indirect tensile strength and uni-axial compressive strength. Fig. 1 shows a schematic of the  $p$ – $q$  space which defines the Mohr–Coulomb line.

The objective of this work is to describe the consolidation behaviour of commercially pure titanium (CP-Ti) powders in the uncoated condition and when coated with nickel–phosphorus (Ni–P) and nickel–boron (Ni–B) separately. The compaction behaviour of the as-received and electroless coated powders, pressed at five different pressures is studied. A combination of Kawakita and Alderborn models has been used here to explain an increase or decrease in relative densities and the green strength of the compacts found experimentally. The Kawakita model [17] is applied over the entire range of compaction pressures, and in combination with the Alderborn [20] and Mohr–Coulomb relationships. Together with the Ohyanne equation, this approach is used to explain the increase in green strength, which is attributed to an increased contribution from plastic deformation.

## 2. Experimental procedure

Two types of hydride–dehydride (HDH) processed commercially pure titanium powder, referred to as grades A and B, have been selected for coating. These two grades differ in their particle shape, size and composition.

The powders were each coated with nickel–phosphorus and nickel–boron layers. Prior to electroless plating, the powders were oven dried at 110 °C for 1 h to remove the moisture present on the surface of the particles. The electroless plating process was carried out in two stages: (1) pre-treatment and (2) plating. The pre-treatment stage is used to activate the particle surface for further metal deposition during the electroless plating stage. Whilst the procedure for nickel–phosphorus (Ni–P) plating on titanium powders is explained in detail elsewhere [29], the procedure for nickel–boron (Ni–B) plating is described briefly below.

The pre-treatment stage for Ni–B coating was prepared with the following composition: nickel chloride – 30 g·l<sup>-1</sup>, sodium hydroxide – 90 g·l<sup>-1</sup>, ethylene diamine – 90 g·l<sup>-1</sup> and sodium saccharin – 0.2 g·l<sup>-1</sup>. Unlike the Ni–P electroless bath, which is an acidic bath, the Ni–B bath is an alkaline bath. To maintain the alkalinity of the bath, sodium hydroxide was added. The pre-treated powders were then transferred to the electroless bath containing nickel chloride – 30 g·l<sup>-1</sup>, sodium borohydride – 0.6 g·l<sup>-1</sup>, sodium hydroxide – 90 g·l<sup>-1</sup>, ethylene diamine – 90 g·l<sup>-1</sup> and sodium saccharin – 0.2 g·l<sup>-1</sup>. The pH and temperature of the electroless bath were maintained at 13 and 90 °C respectively. The coating time maintained during pre-treatment and electroless plating was 1 h for each process.

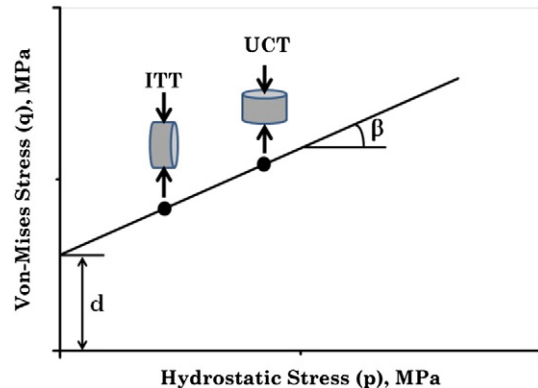


Fig. 1. Mohr–Coulomb line in  $p$ – $q$  space to determine cohesion ( $d$ ) and angle of friction ( $\beta$ ) from indirect tensile test (ITT) and uni-axial compression test (UCT).

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