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Shear deformation with imposed hydrostatic pressure for enhanced compaction of powder

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In this paper equal channel angular extrusion with back pressure was used to compact Ti–6Al–4V powder at 400 °C, achieving relative densities of 98.3–98.6% and green strengths up to 750 MPa. The novelty of the approach arises from the notion that severe shear deformation is an important factor for consolidation. Improved compaction is related to enhanced self-diffusion through the creation of additional diffusion paths (defects) and the imposed hydrostatic pressure. The role of deformation mechanisms in improving compaction is discussed.

Keywords: Ti–6Al–4V powder compaction; Equal channel angular extrusion; Back pressure; Pores; Density

Powder metallurgy (PM) is considered a low-cost production route for near-net-shape components. Pre-alloyed powder stock is normally used to produce compacts with high integrity properties and uniform chemical composition [1]. The main drawback of PM is often the level of residual porosity in the final product, which necessitates a costly hot isostatic pressing (HIP) stage, where relative densities of 98–100% can be obtained at processing temperatures in excess of 800 °C. For Ti–6Al–4V powder, HIP is typically performed at temperatures of 845–955 °C and a pressure of ~105 MPa for 2–4 h. At these temperatures, contamination of powder and compact by gaseous constituents is quite high and, therefore, an inert atmosphere is required.

This research was to investigate the potential for cost-effective, enhanced consolidation of pre-alloyed Ti–6Al–4V powder at temperatures of 400 °C and below using equal channel angular extrusion (ECAE), with applied back pressure [2–4]. This approach could lead to decreases in the manufacturing cost, and avoid the need for expensive atmosphere control. The reasons for enhanced compaction by severe shear deformation with imposed hydrostatic pressure and the physical mechanisms involved were investigated.

A hydride–dehydride (HDH), pre-alloyed Ti–6Al–4V powder was used in this research. The irregularly shaped particles were between 50 and 150 μm in size and had a Vickers hardness of 358 HV. X-ray diffraction (XRD) analysis of the Ti–6Al–4V powder revealed predominantly α-phase, with β-phase being below 5%. Other constituents or contaminants, such as titanium oxide or hydride were below detectable limits.

ECAE with back pressure was performed using a specially designed unit with a heated die, which allowed for isothermal ECAE within the temperature range between 20 and 450 °C. The back pressure is regulated by a horizontal hydraulic cylinder, to maintain the pressure at a preset level between 20 and 500 MPa. The unit is placed in a 1000 kN press which provides the forward pressure of extrusion. A vertical entry channel makes a 90° angle with the horizontal exit channel. The powder was poured directly into the vertical entry channel and during pouring the back pressure punch was positioned deep within the exit channel to contain the powder within the vertical channel. As forward pressure is gradually increased, a significant hydrostatic pressure is created in the vertical channel, pre-compacting the powder. When the pressure exceeds the pre-set back pressure, shear plastic deformation of the pre-compact commences and flow into the exit channel against the back pressure punch occurs.

The role of shear deformation in compaction was assessed firstly by comparing the microstructures of

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material taken before and after the shear line. In addition the hardness in both regions and the green strength of compacts (ASTM B312-96) were determined. A change in the size and distribution of pores after shear deformation was observed optically as shown in Figure 1a. At room temperature, the particles initially pre-compacted under high hydrostatic pressure (to an initial relative density of ~89–91%) had their shape redefined, apparently by brittle fracture of surface protrusions, and underwent realignment upon passing the shear plane. The compact with a relative density of ~95% was achieved at room temperature. Hardness measurements recorded from material before and after the shear plane revealed an approximate doubling of hardness from 152 to 319 HV and 226 to 429 HV for back pressures of 200 and 300 MPa, respectively. However, the green strength of the compacts produced at room temperature was particularly low. As the temperature was increased, plastic deformation of the particles became more significant and interparticle bonding improved. By comparison, the green strength of the compact produced at 400 °C was 450 MPa and 780 MPa for back pressures of 260 MPa and 350 MPa, respectively (Fig. 1b), while the green strength of the compact produced by conventional compression at 400 °C under hydrostatic pressure of 430 MPa was approximately 100 MPa. The relative density under these conditions was 98.3–98.6% (Fig. 1b). The hardness of the compacts produced at 400 °C with back pressures of 260 MPa and 350 MPa was 400 HV and 425 HV, respectively. Using the relationship between hardness and flow curve characteristics developed in [5] for Ti–6Al–4V alloy these hardness values correlate with a yield stress of 1046–1112 MPa and an ultimate tensile stress 1252–1267 MPa. These values are slightly higher than those reported for Ti–6Al–4V compacts produced by the HIP process [6]. However, the direct comparison is difficult as the HIP compaction of PA Ti–6Al–4V is normally done at temperatures of 845–955 °C, for 2–4 h, which leads to softer samples with lower flow curve characteristics than those produced in this research. It will later be shown that the dislocation density in the ECAE processed material is extremely high, and with no annealing possible at processing temperatures as low as 400 °C, a significant hardening effect is expected.

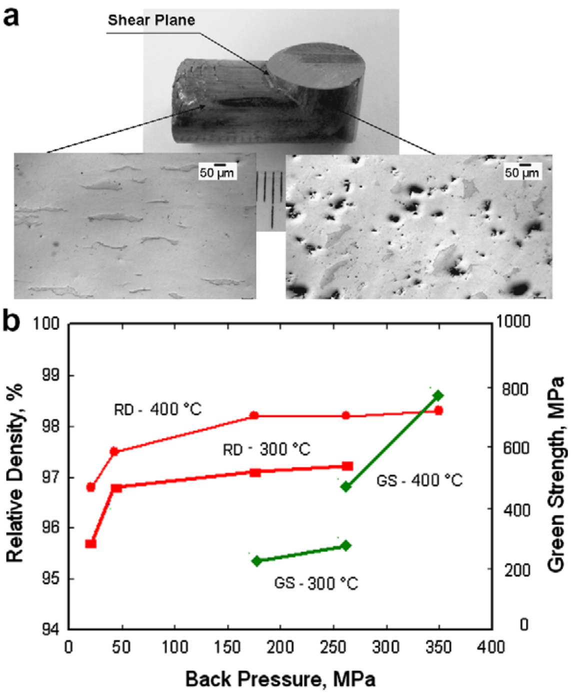

Figure 1. Characteristics of compacts produced by ECAE with back pressure: (a) pore distribution in the compact produced by ECAE at 400 °C and back pressure of 262 MPa before (right) and after (left) shear plane; (b) relative density and green strength versus back pressure for compacts produced by ECAE at 300 and 400 °C.

The increases in strength and relative density achieved by ECAE with back pressure at 400 °C may be attributed to improved self-diffusion at the particle interface due to three factors: increasing contact area between the neighboring particles, temperature and hydrostatic pressure. The temperature and hydrostatic pressure dependence of the self-diffusion coefficient, D, for Ti in α-Ti was calculated using an Arrhenius equation of the form:

$$D = D_0 \exp\left(\frac{-Q}{RT}\right) \tag{1}$$

where: $D_0 = 6.6\times\ 10^{-5}\ \mathrm{cm}^2\ \mathrm{s}^{-1}$ and Q is an activation energy that has components attributable to temperature, $Q_\mathrm{T} = 169.1$ kJ/mol, and hydrostatic pressure, Q_HP [7], where:

$$Q_\mathrm{HP} = \left(P + \sigma_y/\sqrt{3}\right) \cot\left(\frac{\Phi}{2}\right)\Omega \tag{2}$$

Here P is the applied back pressure during ECAE, σ_y is the yield strength of the material at a given temperature T, Φ is the angle of intersection between the entry and exit channels of the ECAE die, and Ω is the atomic volume equal to $3.46 \times 10^{-29}\ \mathrm{m}^3$. The total activation energy Q at a temperature of 400 °C and back pressure of 262 MPa was calculated to be 155.6 kJ mol^{-1}. The diffusion coefficient of Ti in α-Ti at the temperature of 400 °C for zero and 262 MPa of back pressure differs by one order of magnitude, which more than doubles (as shown by the dashed line in Fig. 2) the self-diffusion penetration depth calculated from Ficks' second law of diffusion:

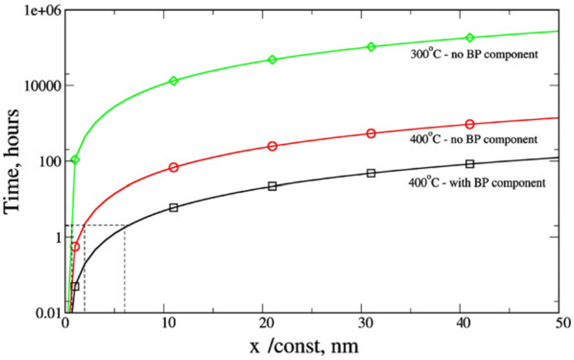

Figure 2. Graph showing the relationship between diffusion time (Ti in α-Ti) and diffusion distance at temperatures of 300 °C and 400 °C at two levels of hydrostatic pressure.