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Use of residual hydrogen to produce CP-Ti powder compacts for low temperature rolling

The present work investigates the optimal level of residual hydrogen in partially de-hydrogenated powder to produce CP-Ti plate compacts using ECAP with back pressure which are subsequently rolled at low temperature. A comparative study of the compaction of two TiH_2 powders and a CP-Ti powder, with particle sizes 150 μm, 50 μm and 45 μm respectively, has been carried out. The hydride powders have also been compacted in a partially de-hydrogenated state. The optimal level of residual hydrogen with respect to the density of the resulting compact and the associated mechanical properties has been defined. ECAP at 300 °C produced compacts from these partially de-hydrogenated powders of 99.5 % theoretical density, while CP-Ti was compacted to almost full theoretical density under the same ECAP conditions. Therefore, the compaction of powder by ECAP does not benefit from temporary hydrogen alloying.

These compacts then were rolled at temperatures ranging from room temperature to 500 °C with an 80 % reduction in a single pass. Heat treatment after the rolling can modify the microstructure to improve the resulting mechanical properties and in this regard the temporary alloying with hydrogen has been observed to offer some significant benefits. It is shown that ECAP followed by low temperature rolling is a promising route to the batch production of fully dense CP-Ti wrought product from powder feedstock that avoids the need to subject the material to temperatures greater than 500 °C. This low temperature route is expected to be efficient from an energy point of view and it also avoids the danger of interstitial contamination that accompanies most high temperature powder processing.

Keywords: CP-Ti powder; TH_2 powder; ECAP compaction; Rolling; Dehydrogenation

1. Introduction

The use of hydrogen for temporary alloying in titanium and its alloys has been demonstrated to lead to improvements in microstructure, properties and processing parameters [1–6]. In particular, the beneficial effect of hydrogen on the compactability and sintering of titanium powders has been investigated [5, 6], for which it is claimed that the use of hydrogenated powder generally facilitates a reduction in the consolidation temperature in the range of 100–140 °C and/or pressure of between 34–67 MPa [7].

Hydrogen induced softening has been used for production of titanium products from chips, swarf and turnings without remelting [8]. The method includes the cold pressing of briquettes from particulate titanium, hydrogenation to a certain level, hot pressing, heat treatment, and de-hydrogenation by vacuum annealing. The mechanical properties of the resulting solid compacts were comparable to those obtained by a conventional molten route followed by hot working.

The consolidation rate of hydride powder was found to be faster than that of commercially pure titanium (CP-Ti) powder and this was also observed to be related to the hydrogen content [7]. The sintering of hydrogenated blended elemental powder has also been observed to occur more readily [9] compared to non-hydrogenated powders. This effect was explained through the role of hydrogen in both protecting the particle surfaces from oxidation and promoting increased chemical activity of the surface.

Hot pressing of titanium hydride rather than hydrogen-free powder has been shown to result in lower porosity parts, in which the porosity reduction was facilitated through de-hydrogenation during the sintering stage. Titanium hydride powder was also used to produce Ti-6Al-4V alloy by mixing a blend of hydrogenated titanium with 10 wt.% of a 60Al-40V master alloy powder. The compacted samples reached 98.5 % and 99.5 % of the theoretical density before and after sintering respectively. In contrast, the corresponding densities of non-hydrogenated powders were 85–88 % before and 90–95 % after sintering [10].

Among the existing powder production processes the hydride/de-hydride method involves a number of processing stages from the embrittlement of a suitable metal billet stock by hydrogen saturation, comminution to powder and then de-hydrogenation to produce the final powder product [11]. For this production process, the elimination of or a reduction in the time/temperature cycle of the last stage to retain a particular level of hydrogen in the powder will lead to cost savings and improvement in the properties of the titanium compacts. The present work investigates the optimal level of residual hydrogen to produce CP-Ti powder compacts using equal channel angular pressing (ECAP) followed by low temperature rolling.

2. Experimental

2.1. Characterisation of as-received powders

The hydride powders, THP-100 and THP-325, and the CP-Ti powder used in this investigation were all sourced from Northwest Institute for Non-ferrous Metal Research (NIN), China. Scanning electron microscopy (SEM) of the loose powders revealed an irregular shape and rough surface for individual particles with the meshed particle size of 150 μm (THP-100), 50 μm (THP-325) and 45 μm (CP-Ti), Fig. 1.

The Vickers micro-indentation hardness of individual particles mounted in epoxy, based on an average of 20 measurements with a standard deviation of 5–10, showed lower values for the Ti hydrides (107–110 HV) compared to CP-Ti (201 HV) due to the brittleness of the THP powders. Microhardness was measured on mounted loose particles according to ASTM, E384–99 standard with a load of 10–25 g and a corresponding average indentation width of 13–20 μm.

X-ray diffraction (XRD) analysis was conducted to determine the constituent phases present in the powders, which were found to be predominantly α-phase for CP-Ti and δ-phase for THP powders, Fig. 2.

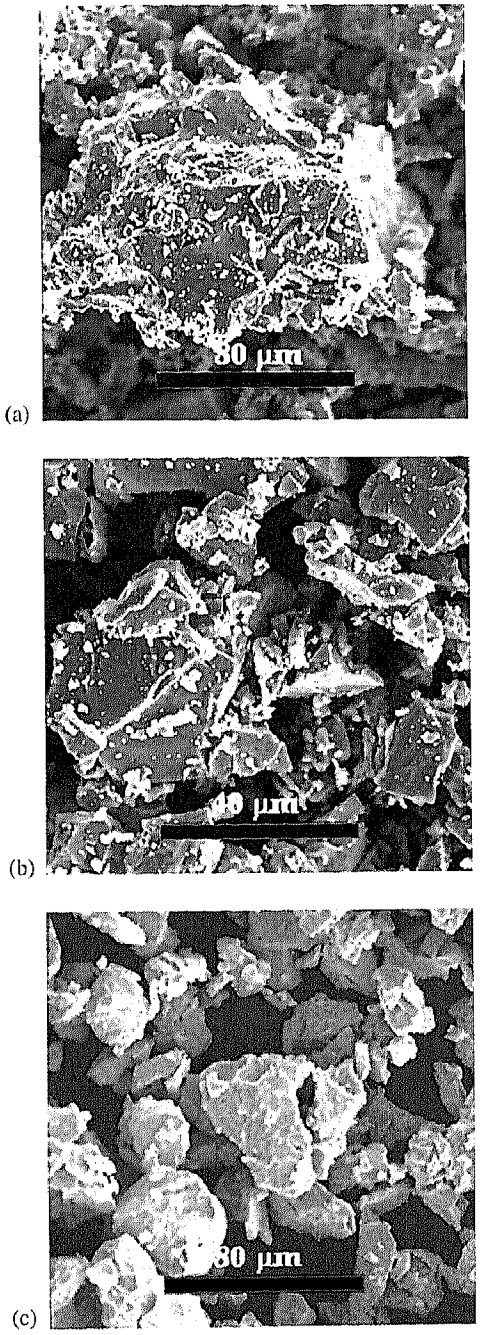


Fig. 1. SEM images of unmounted as-received powder; (a) THP-100; (b) THP-325; (c) CP-Ti.

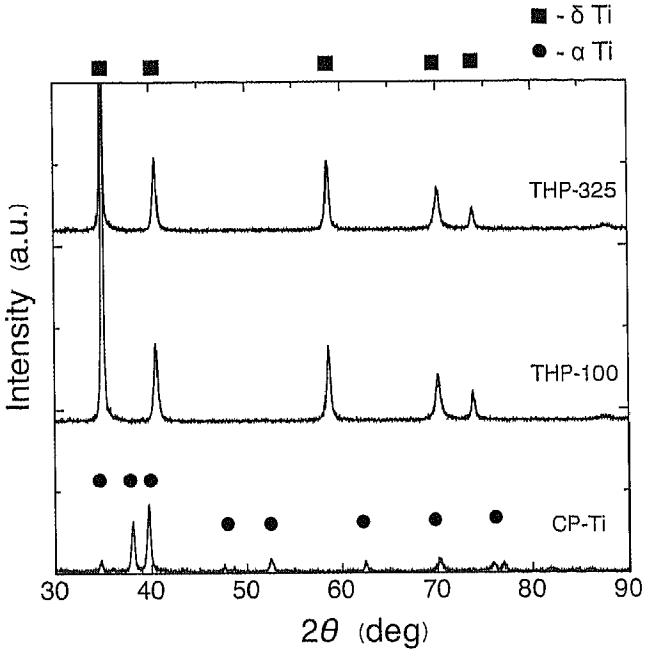
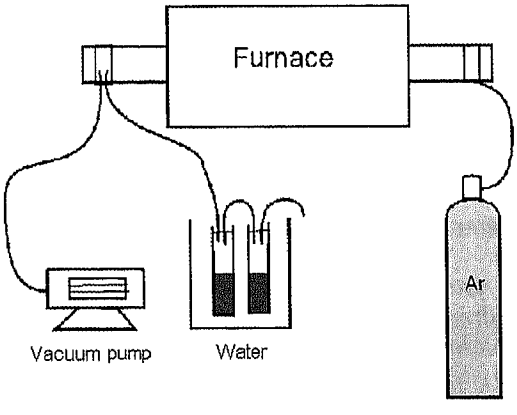


Fig. 2. X-ray diffraction analysis of phases in as-received powders.

2.2. De-hydrogenation

De-hydrogenation of the THP-100 and THP-325 powders was carried out at 700 °C for two and four hours. A schematic of the furnace used for de-hydrogenation is shown in Fig. 3. The decomposition of the hydride particles resulted in CP-Ti powders with different levels of residual hydrogen, which



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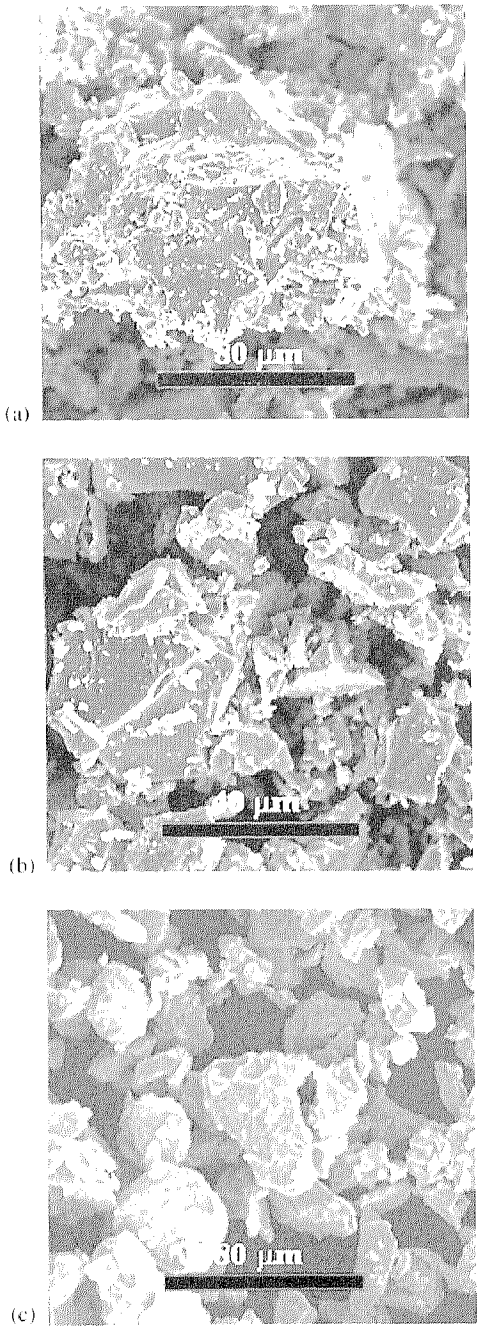

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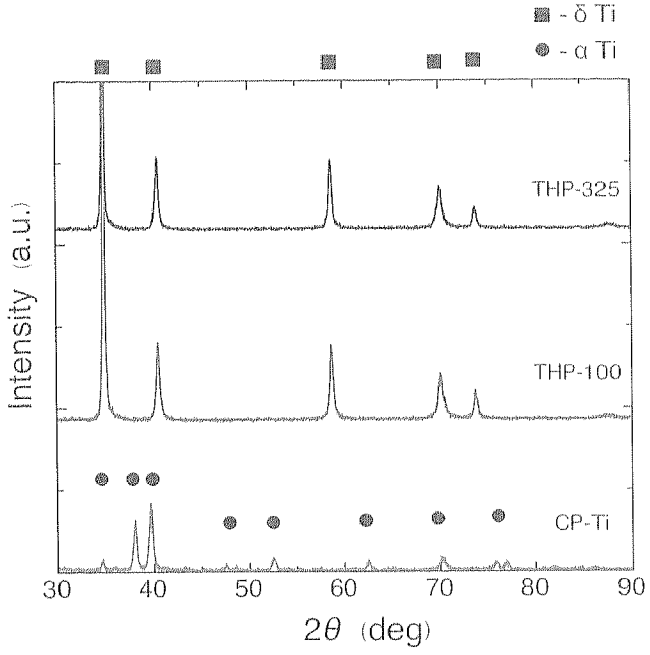

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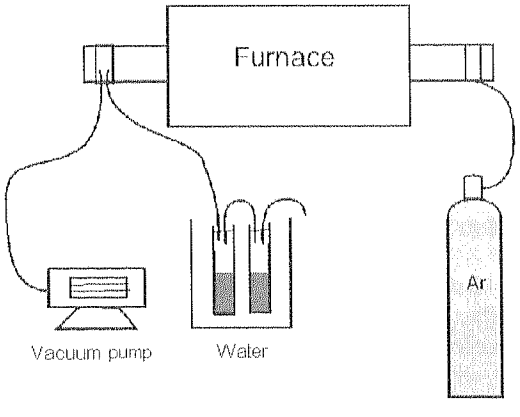

Fig. 3. Schematic of the furnace used for de-hydrogenation of the THP powders.

Table 1. Powders obtained after partial dehydrogenation of hydride-powder.

Designation	Powder	Particle Size (μm)	De-hydrogenation conditions	H (wt.%)	f_δ
THP-$100_{0.61}$	THP-100	150	700 °C, 2 h	0.614	0.36
THP-$100_{0.38}$	THP-100	150	700 °C, 4 h	0.384	0.25
THP-$325_{0.55}$	THP-325	50	700 °C, 2 h	0.547	0.34
THP-$325_{0.46}$	THP-325	50	700 °C, 4 h	0.461	0.28

were determined by a LECO analyser (see Table 1). The equilibrium ratios for the α and δ-phases can be estimated using the Ti-H phase diagram [12], Fig. 4, and the values thus obtained are included in Table 1.

In addition, a complete de-hydrogenation heat treatment was performed on the rolled plates, which were made from the partially dehydrogenated powders. The final heat treatment was conducted in the same furnace at 500 °C for 1 h, which not only removes the remnant hydrogen content but also retains a fine grained microstructure as shown in [13, 14].

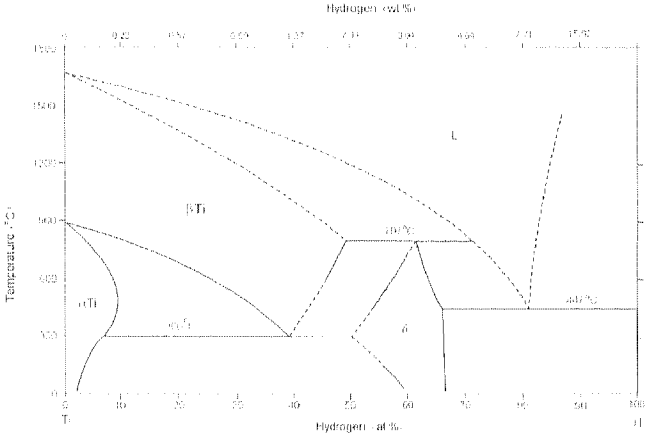

Fig. 4. Ti-H phase diagram (from [12]).

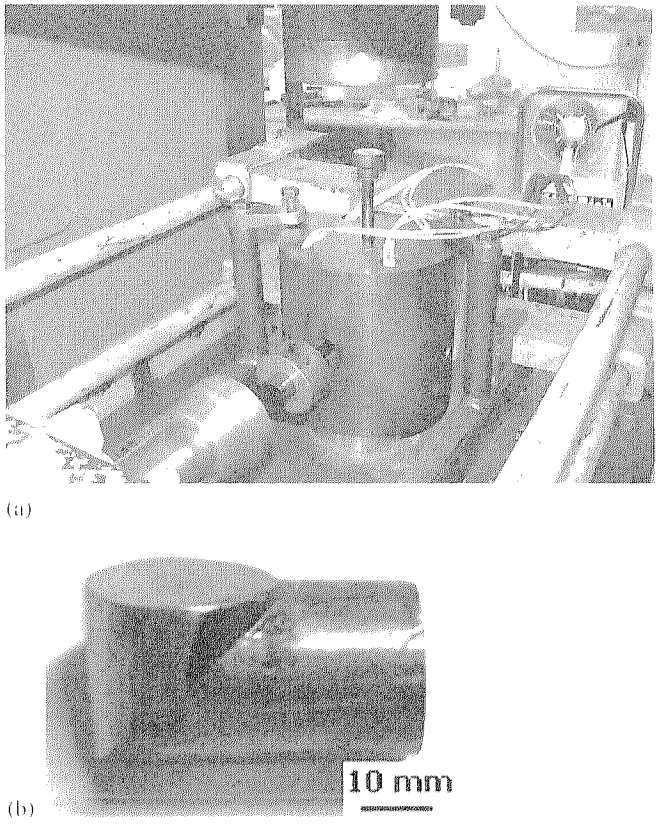

Fig. 5. (a) Unit for elevated-temperature ECAP with back-pressure and (b) a sample compacted from THP-100 powder.

2.3. ECAP of powder

All the powders were compacted by ECAP with back pressure using a specially designed rig, Fig. 5, at temperatures varying from room temperature to 300 °C. The back-pressure was maintained by a horizontal hydraulic cylinder at a preset level of 260 MPa. The unit was placed in a 1000 kN press which provided the forward pressure for extrusion. The diameters of both channels in the ECAP die are 10 mm with the length of the entry channel and exit channels equal to 75 mm and 35 mm, respectively. The 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 loose powder within the vertical channel. The amount of powder charge was determined by the volume of entry channel filled up to the hight of 70 mm with a tap density of 20 %. As the applied force is increased gradually on the extrusion punch, a large hydrostatic pressure is created in the vertical channel pre-compacting the powder. When the pressure exceeds the pre-set backward pressure, shear plastic deformation of the pre-compact commences and it flows into the exit channel against the back pressure punch.

ECAP was performed with a constant speed of 0.5 mm s^{-1}. Graphite spray was used to lubricate the die walls. Resistance heating was used to raise the temperature of the die to the pre-set temperature over a period of about 5 min and this was followed by a 5 min soak prior to extrusion.

2.4. Density measurements

The density of compacts was determined by Archimedes' method (ASTM, B311-93) using tetrachlorethylene, with a density of $\rho_{tetr} = 1.62$ g cm^{-3}, as the immersion liquid:

$$\rho = \frac{W_{air}}{(W_{air} - W_{tetr})}\rho_{tetr} \qquad (1)$$

where W_{air} is the weight of the sample in air, W_{tetr} is weight of the sample in tetrachlorethylene. After calculations the densities were compared to theoretical densities of metals,

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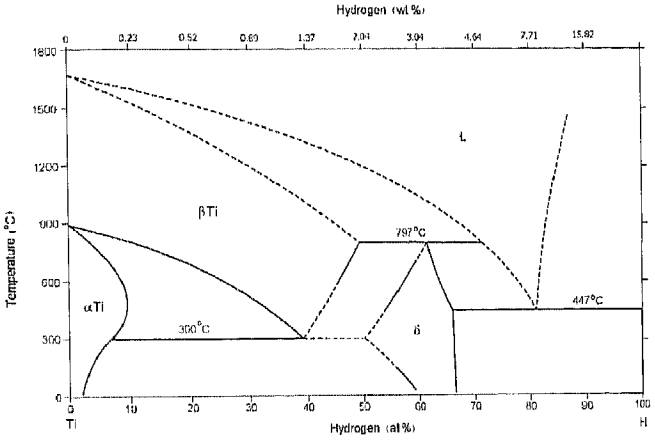

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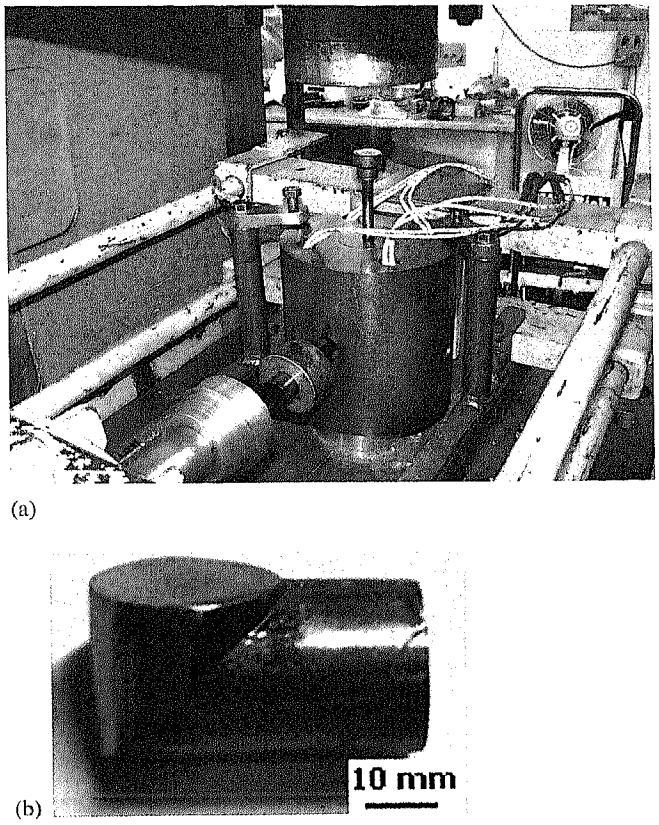

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ρ_t, where $\rho_t = 4.510$ g cm^{-3} has been used for CP-Ti and $\rho_t = 3.72$ g cm^{-3} for hydrides.

2.5. Microstructure characterisation

The powder morphology, deformation mechanisms and microstructural evolution were analysed using optical microscopy (OM), SEM (JEOL JSM-6300F at 15 kV) and transmission electron microscopy (TEM; Philips CM20 at 200 kV). Metallographic sample preparation consisted of grinding on SiC paper, polishing with diamond, and etching with Kroll's reagent (5 % nitric acid, 10 % HF, 85 % water). TEM samples in the form of 3 mm diameter discs were cut from the compact and electro-polished in a twin-jet TENUPOL 5 operating at −50 °C using a solution of 5 wt.% perchloric acid in 95 wt.% methanol. The XRD analysis was performed using a Philips diffractometer equipped with a Cu anode at 40 kV and 25 mA.

2.6. Rolling and mechanical testing

Compacted samples were rolled at temperatures from 500 °C down to RT using a four-high mill (70 mm diameter working rolls) in five passes and using a two-high mill (350 mm diameter rolls) in one pass with a total thickness reduction of 80 %. The rolling temperature was decreased in order to find the lowest possible processing temperature to maintain the integrity and quality of the finished rolled sheet. Tensile tests were conducted using a screw driven Instron at a strain rate of 0.01 s^{-1}. Shear punch tests (SPT) [15] were performed to evaluate the mechanical properties of the extruded billets with the different levels of hydrogen. A punch with radius, r, of 1.5 mm was used with radial clearance set to be 0.1 mm. Samples of 0.3 mm thickness, t, and 10 mm in diameter were cut from the central plane of the compacts. The punch speed was 0.5 mm min^{-1}. From load, P, and displacement, d_f, and known friction force $F = 100$ N, the effective stress and strain were calculated as follows:

$$\sigma_{eff} = \frac{P - F}{2\pi r t} \tag{2}$$

$$\varepsilon_{eff} = \frac{d_f}{t} \tag{3}$$

The strength of the compacts was estimated from expression, [16]: $\sigma_{eff\ max} = 0.62\ \sigma_{UTS}$.

The green strength of the compacts was measured according to ASTM B 312-96 using a three-point bend test. Green strength is defined as the stress required to fracture a specimen, and is calculated using Eq. (4). The apparatus and test specimen were scaled down by a ratio of 1:3 in the present case, with the dimensions of the specimen equal to 10.5 × 4.2 × 2.1 mm, and the span being 8.6 mm.

$$S = \frac{3PL}{2t^2 w} \tag{4}$$

where S – green strength (MPa); P – force required to rupture (N); L – length between support (mm); w – width of specimen (mm); and t – thickness of specimen (mm). The samples were cut parallel to the extrusion direction.

3. Results and discussion

3.1. Compaction prior to rolling

3.1.1. Relative density of compacts

To calculate the relative density of the compacts produced from partially de-hydrogenated powders a rule of mixtures has been used:

$$\rho = \frac{\rho_\alpha f_\alpha + \rho_\delta f_\delta}{f_\alpha + f_\delta} \tag{5}$$

The calculated values coincided very well with data published in [17]. The theoretical density of hydrogenated and de-hydrogenated titanium as function of hydrogen content is shown in Fig. 6.

Initially the compaction of Ti hydrides by ECAP was studied at different temperatures and compared with the compaction of CP-Ti powder under similar conditions. The compaction of Ti hydrides did not require high punch forces. However, the resulting compacts had a very low green strength and could be easily broken during extraction from the ECAP die. The relative density of compacts increased with ECAP temperature regardless of the initial particle size and was close to 99.5 % at 300 °C, Fig. 7. The density of compacts produced from CP-Ti powder reached the theoretical density at 300 °C.

Following these experiments, compacts were produced from the partially de-hydrogenated powders listed in Table 1 using ECAP with 260 MPa of back pressure at different temperatures. The relative densities of these samples were measured and compared with relative densities of the previous specimens and these are shown in Fig. 8.

An increase in the relative densities with ECAP temperature is seen for all four powders, Fig. 8a and b. The best compaction was obtained at 300 °C. The level of hydrogen

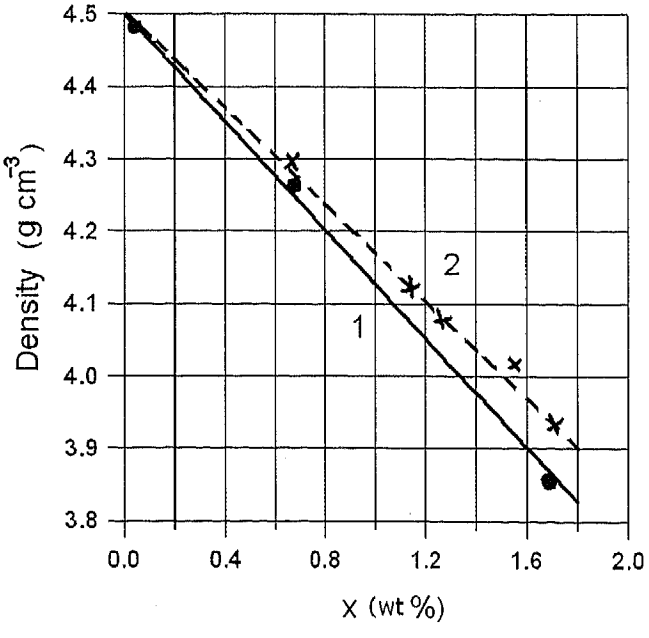

Fig. 6. Effect of hydrogen content, x wt.%, of titanium on density (± 0.03 g cm^{-3}) of hydrogenated titanium (1) and titanium dehydrogenated (2) (from [17]).

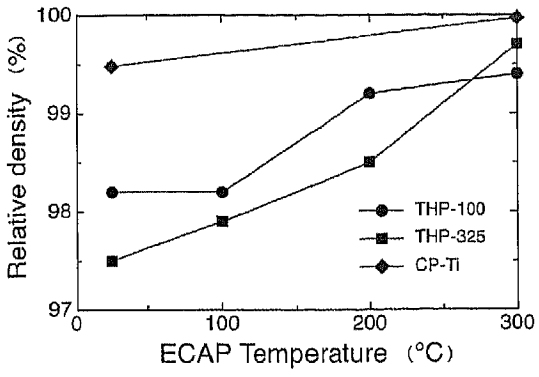

Fig. 7. Relative density vs. ECAP temperature for hydrides and CP-Ti powders.

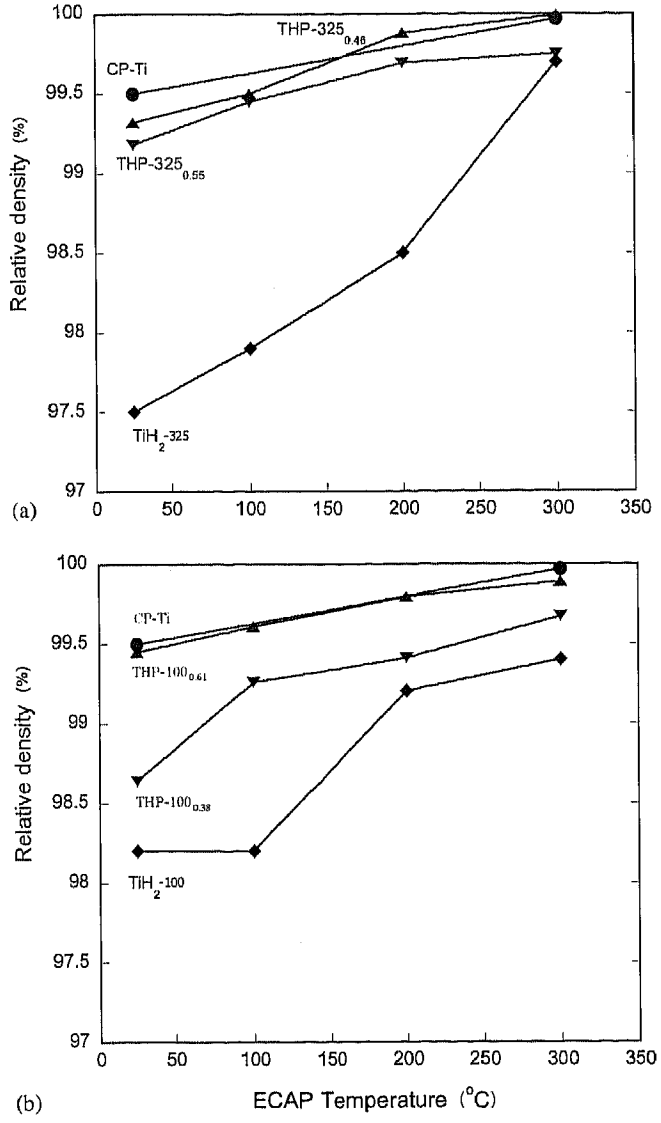

Fig. 8. Relative density of compacts produced from hydrates, partially dehydrogenated powders and CP-Ti vs. ECAP temperature; (a) THP-325; (b) THP-100.

for which the relative densities of the compacts produced from the de-hydrogenated powder was close to 99.9 %, was dependent on the particle size. It was 0.46 wt.% for THP-325 and 0.61 wt.% for THP-100. This difference probably arises from the difference in hydrogen diffusion distances [18] but the exact mechanism is not known.

3.1.2. Hardness of compacts

The hardness of the compacts decreased very slightly with ECAP temperature for partially de-hydrogenated powders, but for CP-Ti the drop in hardness was about 30 VHN, Fig. 9.

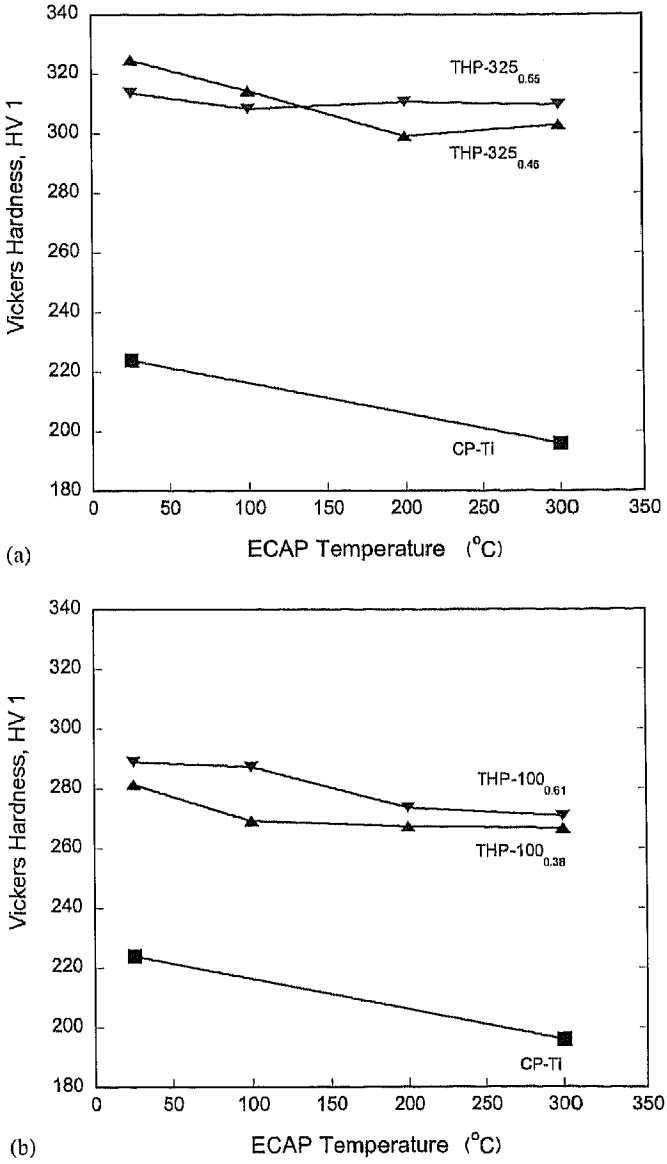

Fig. 9. Vickers Hardness of compacts produced from partially dehydrogenated powders and CP-Ti vs. ECAP temperature; (a) THP-325; (b) THP-100.

3.1.3. Optical microscopy

No porosity in the compacts was observed by optical microscopy, regardless of the level of hydrogen, when the density was higher than 99.8 %. Only a very few small pores were found in the THP-$325_{0.55}$ compact with a density of about 99.5 %. The typical arrangement of particles parallel to the shear plane can be seen in Fig. 10 where the structures of compacts from THP-$100_{0.61}$, THP-$325_{0.46}$ and CP-Ti powders are shown.

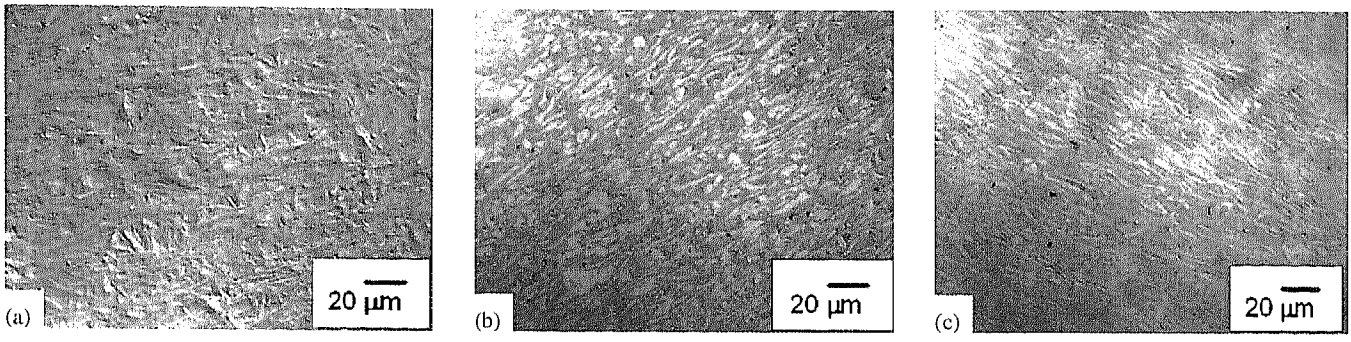

Fig. 10. Optical micrographs of compacts produced from partially de-hydrogenated and CP Ti powders using ECAP with 350 MPa back pressure and temperature of 300 °C; (a) THP-$100_{0.61}$; (b) THP-$325_{0.46}$; (c) CP Ti.

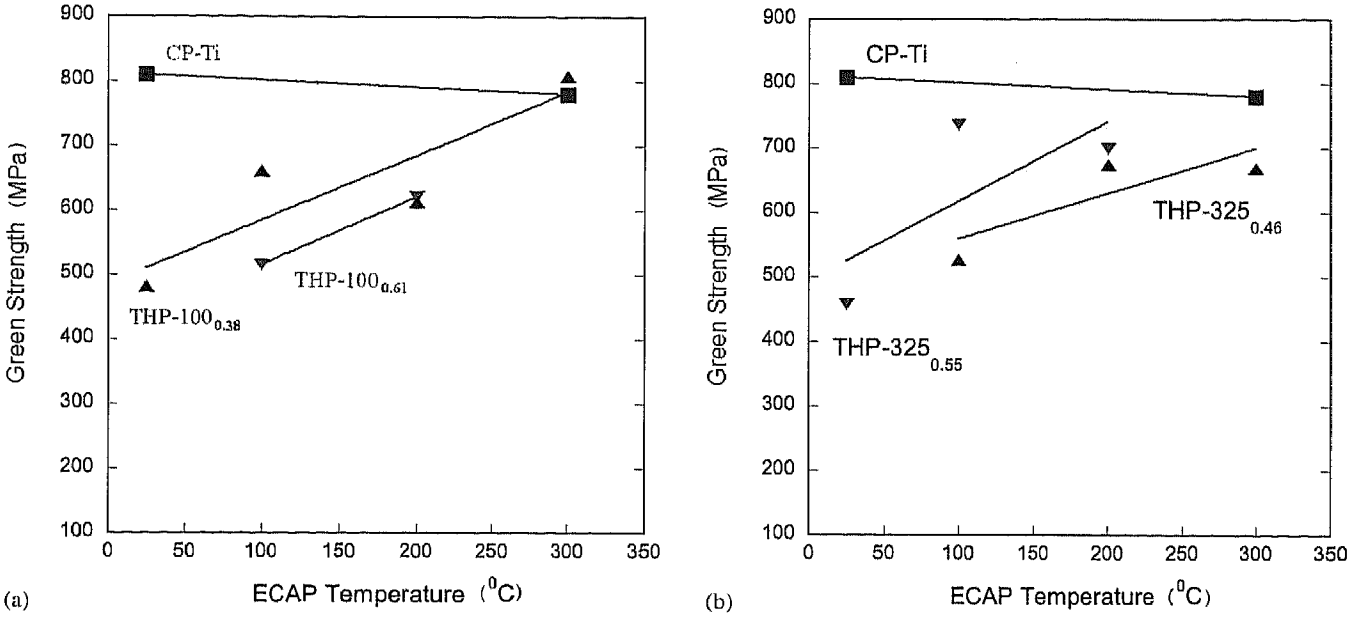

Fig. 11. Green strength of compacts produced from partially dehydrogenated powders and CP-Ti vs. ECAP temperature; (a) THP-325; (b) THP-100.

Table 2. Mechanical properties of compacts produced from hydrogenated powder.

No	Powder Designation	ECAP Temperature (°C)	Strength (Shear punch test) (MPa)	Effective Strain (Shear punch test) (%)
1	CP-Ti	100	299	3
2	CP-Ti	300	303	31
3	THP-$100_{0.61}$	RT	251	0.6
4	THP-$100_{0.61}$	100	239	1
5	THP-$100_{0.61}$	200	324	2
6	THP-$100_{0.61}$	300	367	5
7	THP-$100_{0.38}$	RT	327	2.6
8	THP-$100_{0.38}$	100	366	5
9	THP-$100_{0.38}$	200	394	14
10	THP-$100_{0.38}$	300	378	29
11	THP-$325_{0.55}$	RT	338	1
12	THP-$325_{0.55}$	100	228	2.6
13	THP-$325_{0.55}$	200	288	3.1
14	THP-$325_{0.55}$	300	305	9
15	THP-$325_{0.46}$	RT	338	1
16	THP-$325_{0.46}$	100	399	1.1
17	THP-$325_{0.46}$	200	396	4.6
18	THP-$325_{0.46}$	300	383	17

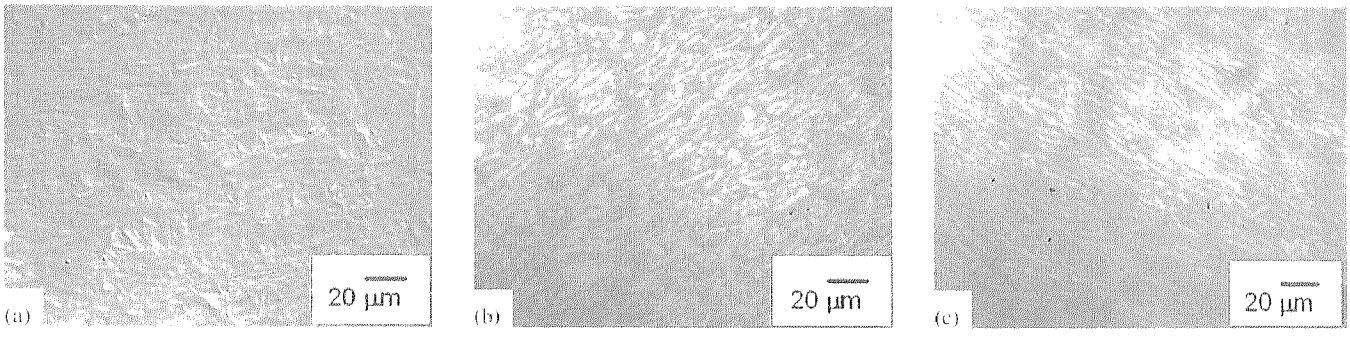

Fig. 10. Optical micrographs of compacts produced from partially de-hydrogenated and CP Ti powders using ECAP with 350 MPa back pressure and temperature of 300 °C; (a) THP-$100_{0.61}$; (b) THP-$325_{0.46}$; (c) CP Ti.

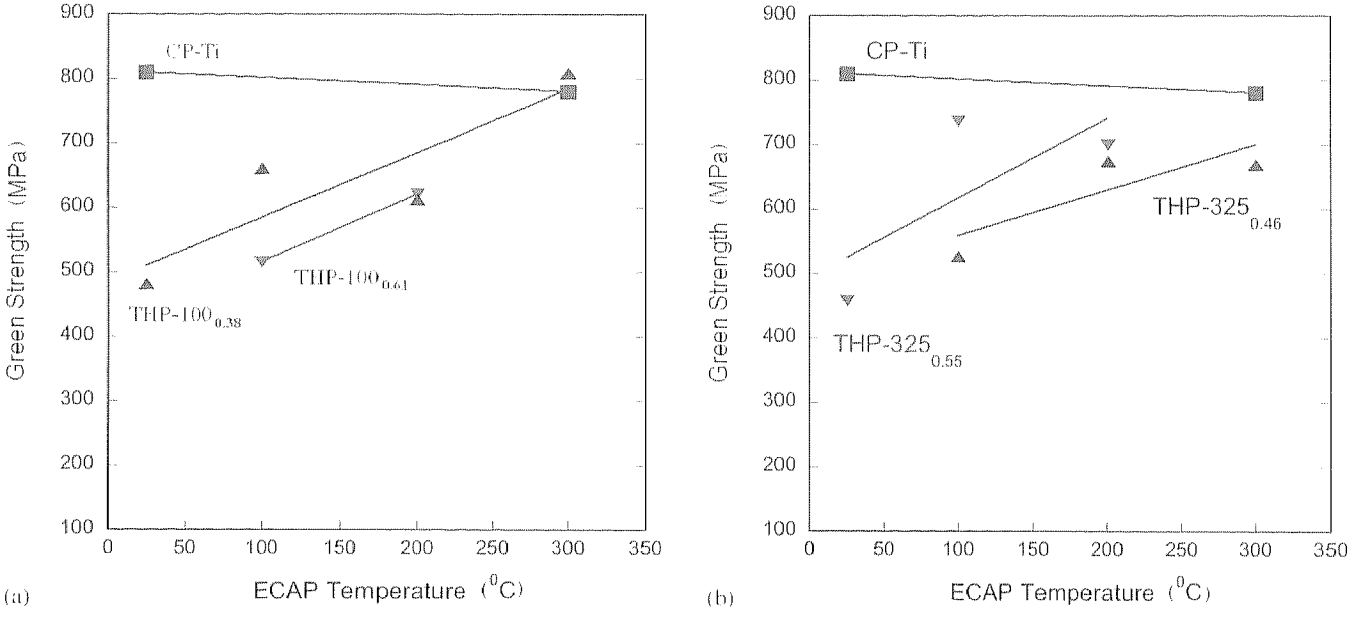

Fig. 11. Green strength of compacts produced from partially dehydrogenated powders and CP-Ti vs. ECAP temperature; (a) THP-325; (b) THP-100.

Table 2. Mechanical properties of compacts produced from hydrogenated powder.

No	Powder Designation	ECAP Temperature (°C)	Strength (Shear punch test) (MPa)	Effective Strain (Shear punch test) (%)
1	CP-Ti	100	299	3
2	CP-Ti	300	303	31
3	THP-$100_{0.61}$	RT	251	0.6
4	THP-$100_{0.61}$	100	239	1
5	THP-$100_{0.61}$	200	324	2
6	THP-$100_{0.61}$	300	367	5
7	THP-$100_{0.38}$	RT	327	2.6
8	THP-$100_{0.38}$	100	366	5
9	THP-$100_{0.38}$	200	394	14
10	THP-$100_{0.38}$	300	378	29
11	THP-$325_{0.55}$	RT	338	1
12	THP-$325_{0.55}$	100	228	2.6
13	THP-$325_{0.55}$	200	288	3.1
14	THP-$325_{0.55}$	300	305	9
15	THP-$325_{0.46}$	RT	338	1
16	THP-$325_{0.46}$	100	399	1.1
17	THP-$325_{0.46}$	200	396	4.6
18	THP-$325_{0.46}$	300	383	17

3.1.4. Green strength

Green strength of the compacts produced from partially de-hydrogenated powder increased with temperature of ECAP, Fig. 11. This can be explained by the continuous change in hydrogen content during ECAP and partial dissolution of some hydrides. This effect is not present in compacts produced from CP-Ti powder and as can be seen, the green strength of those compacts only slightly depends on the temperature of ECAP.

3.1.5. Shear punch test

Shear punch tests were performed to estimate the strength and ductility of all compacts and evaluate the possibility of low temperature rolling. The results are presented in Table 2.

The ductility of the compacts increases with temperature of ECAP compaction and a maximum value for each of the powders was observed when ECAP was performed at 300 °C. For all the samples compacted at 300 °C the ductility was inversely proportional to the volume fraction of δ-phase, f_δ, see Table 1.

3.2. Rolling of compacted powder billets

Samples for subsequent rolling were chosen after consideration of the best compactability of the powder, which were represented by the compacts produced from THP-$100_{0.61}$ and THP-$325_{0.46}$ powders, with levels of hydrogen content of 0.61 wt.% and 0.46 wt.% respectively.

The compacts produced from CP-Ti and the partly de-hydrogenated hydride powders were rolled at temperatures of 500 °C, 300 °C, 200 °C, 100 °C and RT, to define the lowest possible temperature for rolling whilst maintaining the integrity and quality of the sheet. The rolling parameters for all powders are given in Table 3.

Rolling of compacts produced from CP-Ti powder was conducted initially in multiple passes with intermediate reheating between each pass to minimise cracking. However, it was found that the ductility of the compacts was high enough to permit rolling to a reduction of 80 % in a single pass without loss of integrity. This also held true for compacts produced from partially de-hydrogenated powders. The repeated reheating of the samples between multiple rolling passes also led to a slightly coarser grain size, Fig. 12. Therefore, the majority of the rolling for the partially de-hydrogenated powder compacts was conducted in a single pass.

Table 3. Rolling parameters for compacts produced from partially de-hydrogenated powder.

No	Powder Designation	Rolling Temperature (°C)	Number of Passes
1	CP-Ti	500	5
2	CP-Ti	500	1
3	CP-Ti	300	1
4	CP-Ti	200	1
5	CP-Ti	100	1
6	CP-Ti	RT	1
1a	THP-$100_{0.61}$	500	1
2a	THP-$100_{0.61}$	300	1
3a	THP-$100_{0.61}$	200	1
4a	THP-$100_{0.61}$	100	1
5a	THP-$100_{0.61}$	RT	1
1b	THP-$325_{0.46}$	500	1
2b	THP-$325_{0.46}$	300	1
3b	THP-$325_{0.46}$	200	1
4b	THP-$325_{0.46}$	100	1
5b	THP-$325_{0.46}$	RT	1

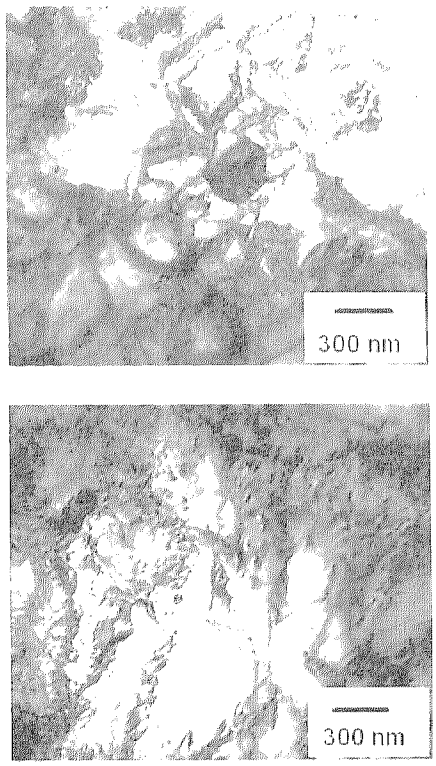

Fig. 12. TEM images of CP-Ti compacts rolled at 500 °C (a) – 1 pass; and (b) – 5 passes.

3.2.1. Optical images of rolled CP-Ti compacts

The microstructures of the compacts rolled at different temperatures are shown in Fig. 13. A typical elongated fibrous rolling structure is seen for all temperatures of rolling.

3.2.2. SEM images of rolled CP-Ti compacts

SEM images of the rolled structures showed that the bonding between particles can be destroyed by intensive metal flow during rolling at room temperature, Fig. 14a. With increasing rolling temperature, the bonding is strong enough to withstand the severe deformation and if the rolling is performed at 300 °C or higher temperature then complete welding between individual powder particles occurs, Fig. 14c.

3.2.3. Mechanical properties of rolled CP-Ti compacts

Tensile properties and density of plates rolled from CP-Ti compacts at 500 °C in 1 and 5 passes are compared to the properties of bulk CP-Ti rod of the same diameter, rolled under the same conditions, see Table 4. It can be seen that there is no significant difference in the properties of the samples regardless of the number of rolling passes. The tensile strength of samples rolled from compacted powder is

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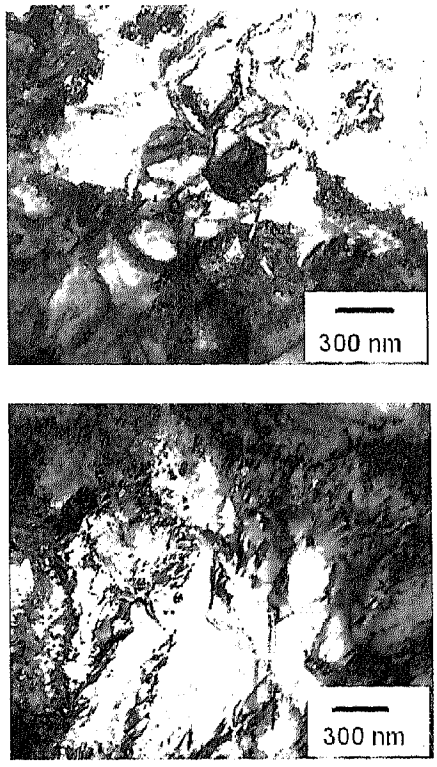
4	CP-Ti	200	1
5	CP-Ti	100	1
6	CP-Ti	RT	1
1a	THP-$100_{0.61}$	500	1
2a	THP-$100_{0.61}$	300	1
3a	THP-$100_{0.61}$	200	1
4a	THP-$100_{0.61}$	100	1
5a	THP-$100_{0.61}$	RT	1
1b	THP-$325_{0.46}$	500	1
2b	THP-$325_{0.46}$	300	1
3b	THP-$325_{0.46}$	200	1
4b	THP-$325_{0.46}$	100	1
5b	THP-$325_{0.46}$	RT	1



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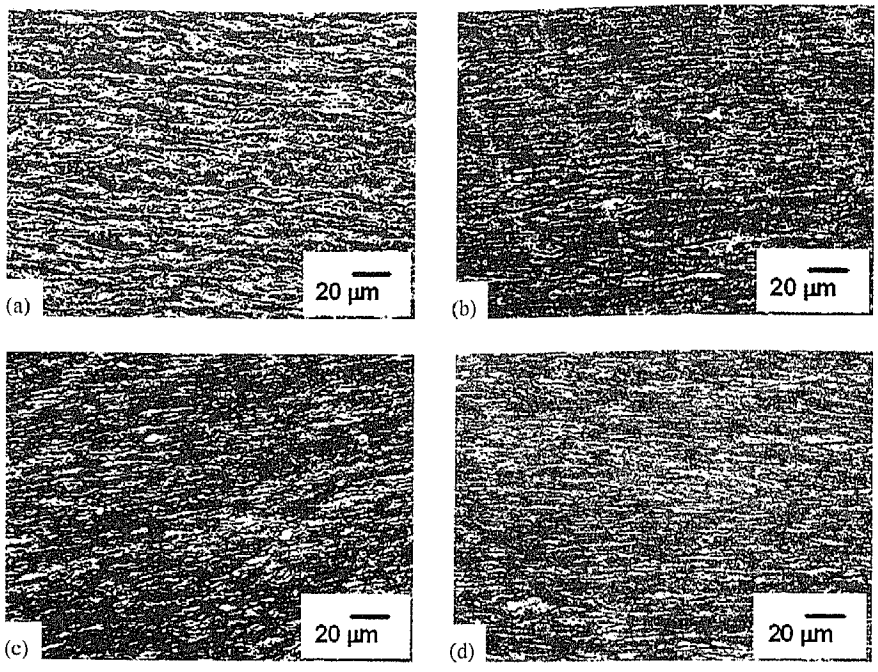


Fig. 13. Microstructure of CP-Ti compacts rolled at different temperatures: (a) 500 °C; (b) 300 °C; (c) 100 °C; (d) RT.

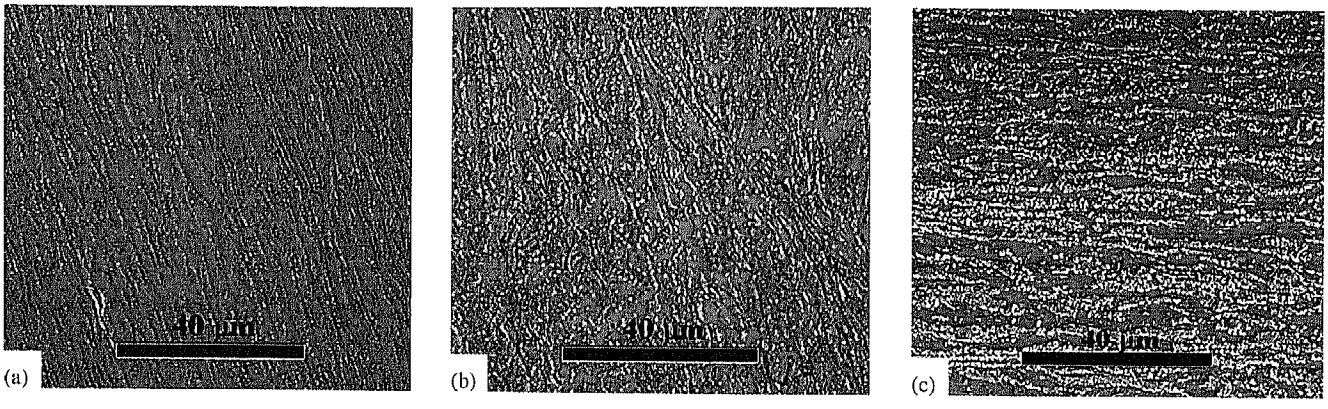


Fig. 14. SEM images of CP Ti compacts rolled at different temperatures (1 pass): (a) RT; (b) 200 °C; (c) 500 °C.

Table 4. Tensile properties of CP-Ti compact rolled at 500 °C.

Processing Parameters	Density ($g\,cm^{-3}$)	Tensile Strength (MPa)	Ductility (%)
ECAP (powder) at 300 °C → Rolling 1 pass at 500 °C	98.3	630	21
ECAP (powder) at 300 °C → Rolling 5 passes at 500 °C	99.0	634	15
Bulk samples → Rolling 1 passes at 500 °C	99.2	691	19
Bulk samples → Rolling 5 passes at 500 °C	99.1	680	18

lower than of that rolled bulk rod by ~45–60 MPa with a similar level of ductility.

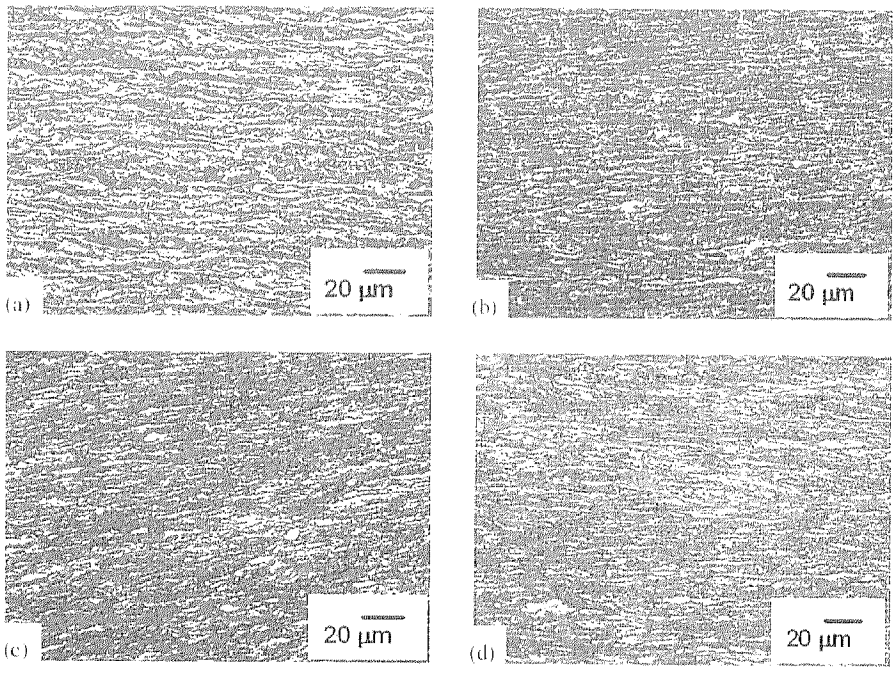
Strength and ductility in shear punch tests for plates rolled at different temperatures are given in Table 5. Comparably high levels of ductility and strength were obtained at rolling temperatures between 100 – 300 °C while at room temperature the strength is 100 MPa lower.

Table 5. Mechanical properties of rolled CP-Ti compacts from shear punch testing.

No	Rolling Temperature (°C)	Strength (Shear punch test) (MPa)	Effective Strain (Shear punch test) (%)
1	RT	324	32
2	100	424	36
3	200	436	38
4	300	402	37
5	500 (1p)	392	31
6	500 (5p)	394	22

3.2.4. Shear punch test of rolled THP-$100_{0.61}$ compacts before and after heat treatment

As can be seen from Table 6, the low temperature de-hydrogenation treatment does not recover ductility, except when the rolling was done at 500 °C. However, it does increase strength. This heat treatment was selected following the results published in [13]. There were no attempts made



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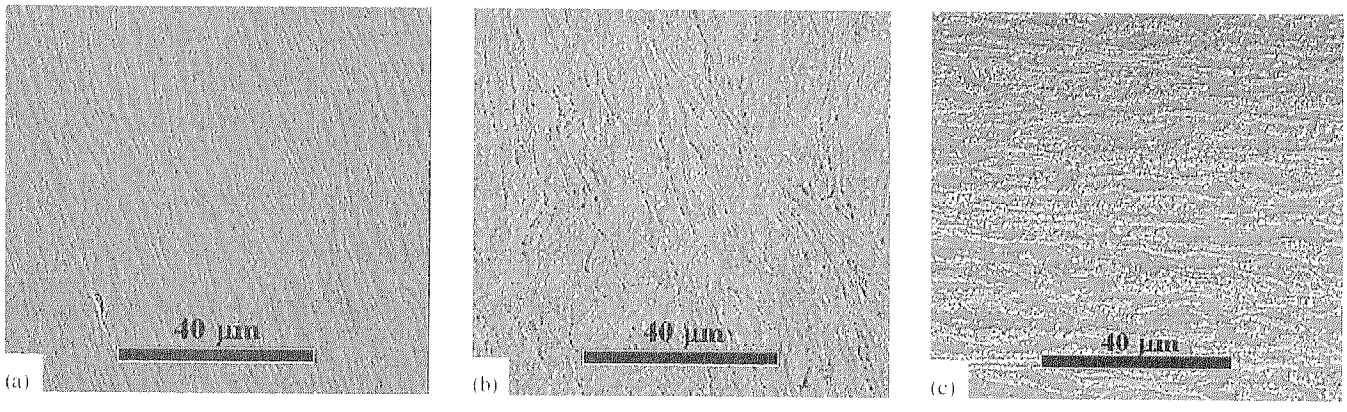

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No	Rolling Temperature (°C)	Before Heat Treatment		After Heat Treatment	
		Strength (MPa)	Effective Strain (%)	Strength (MPa)	Effective Strain (%)
1	RT	242	7.2	388	3.7
2	100	331	6.1	377	5.8
3	200	336	12.3	389	8.2
4	300	382	18.3	425	15.4
5	500	491	4.9	436	30.3

Table 7. Mechanical properties of rolled THP-$325_{0.46}$ compacts from shear punch testing.

No	Rolling Temperature (°C)	Before Heat Treatment		After Heat Treatment	
		Strength (MPa)	Effective Strain (%)	Strength (MPa)	Effective Strain (%)
1	RT	282	7.9	365	11.5
2	100	456	8.2	238	14.7
3	200	512	53.1	412	18.3
4	300	420	11.8	448	23.8
5	500	531	11.2	492	44.5

to optimise the de-hydrogenation parameters to obtain the best properties and microstructure, which is the subject of a separate research program.

3.2.5. Optical images of rolled THP-$100_{0.61}$ compacts before and after de-hydrogenation treatment

Comparison of the microstructure of the sheets rolled at different temperatures before and after the de-hydrogenation treatment, shows that the elongated deformed structure changes to a more recrystallised microstructure with an increase in rolling temperature, Fig. 15. The most uniform microstructure was obtained when rolling was conducted at 500°C and the best strength and ductility, after the de-hydrogenation treatment, were also observed at this temperature.

3.2.6. Shear punch test of rolled THP-$325_{0.46}$ compacts before and after heat treatment

In contrast to the THP-$100_{0.61}$ powder, the low temperature de-hydrogenation treatment recovers ductility and in some cases improves strength of rolled THP-$325_{0.46}$ powder compacts, except when rolling was conducted at 200°C, Table 7.

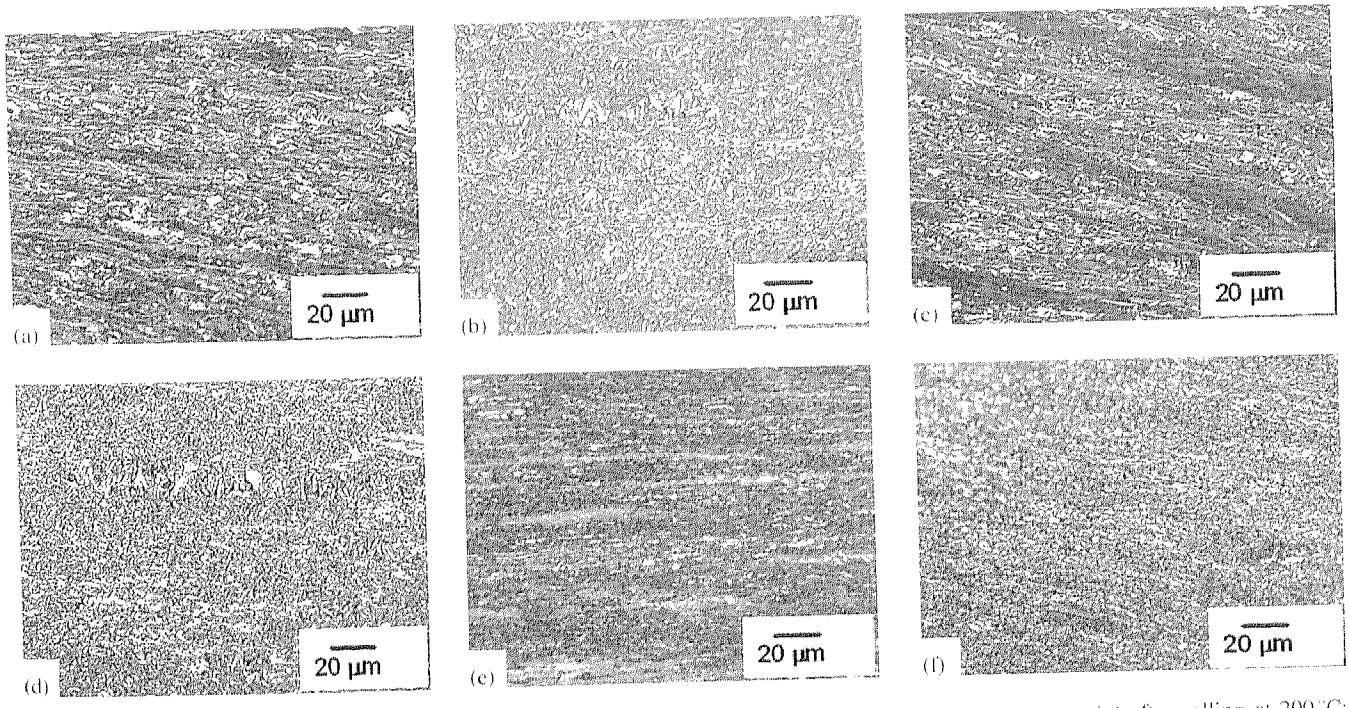

Fig. 15. Microstructure of plates rolled from THP-$100_{0.61}$ compacts before and after de-hydrogenation treatment: (a) after rolling at 200°C; (b) after rolling at 200°C followed by de-hydrogenation; (c) after rolling at 300°C; (d) after rolling at 300°C followed by de-hydrogenation; (e) after rolling at 500°C; (f) after rolling at 500°C followed by de-hydrogenation.

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		Strength (MPa)	Effective Strain (%)	Strength (MPa)	Effective Strain (%)
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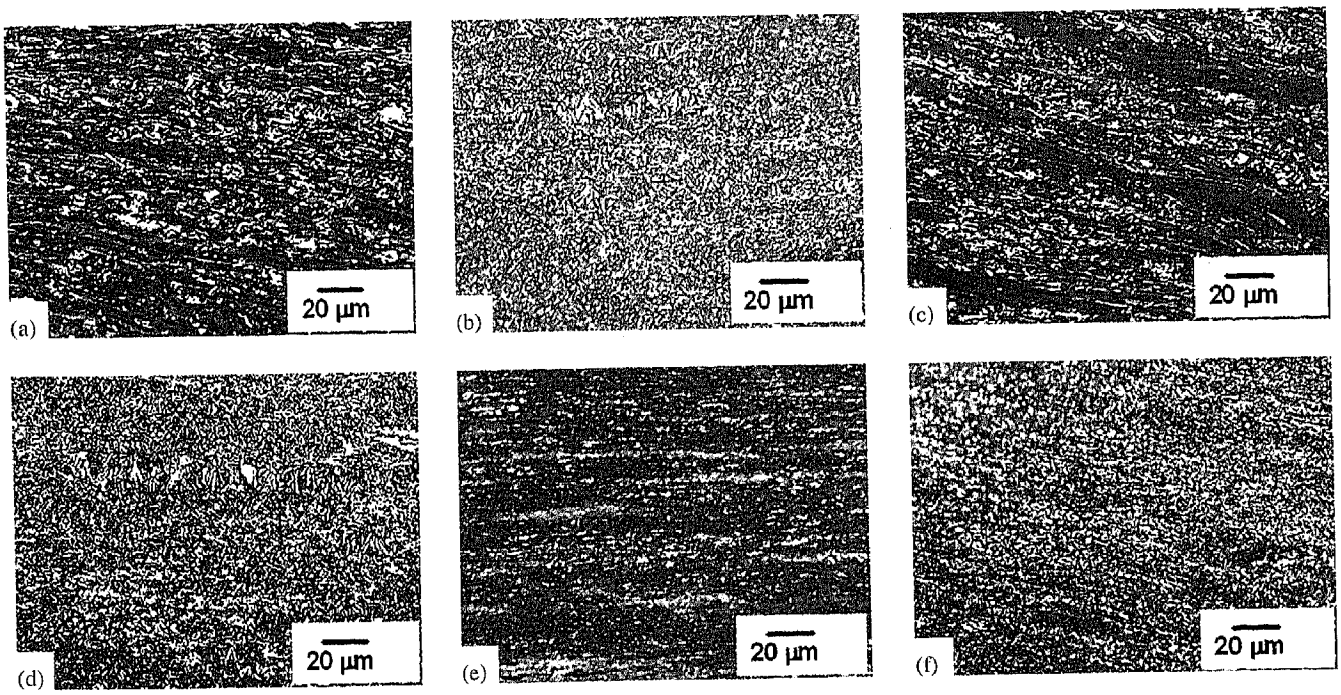

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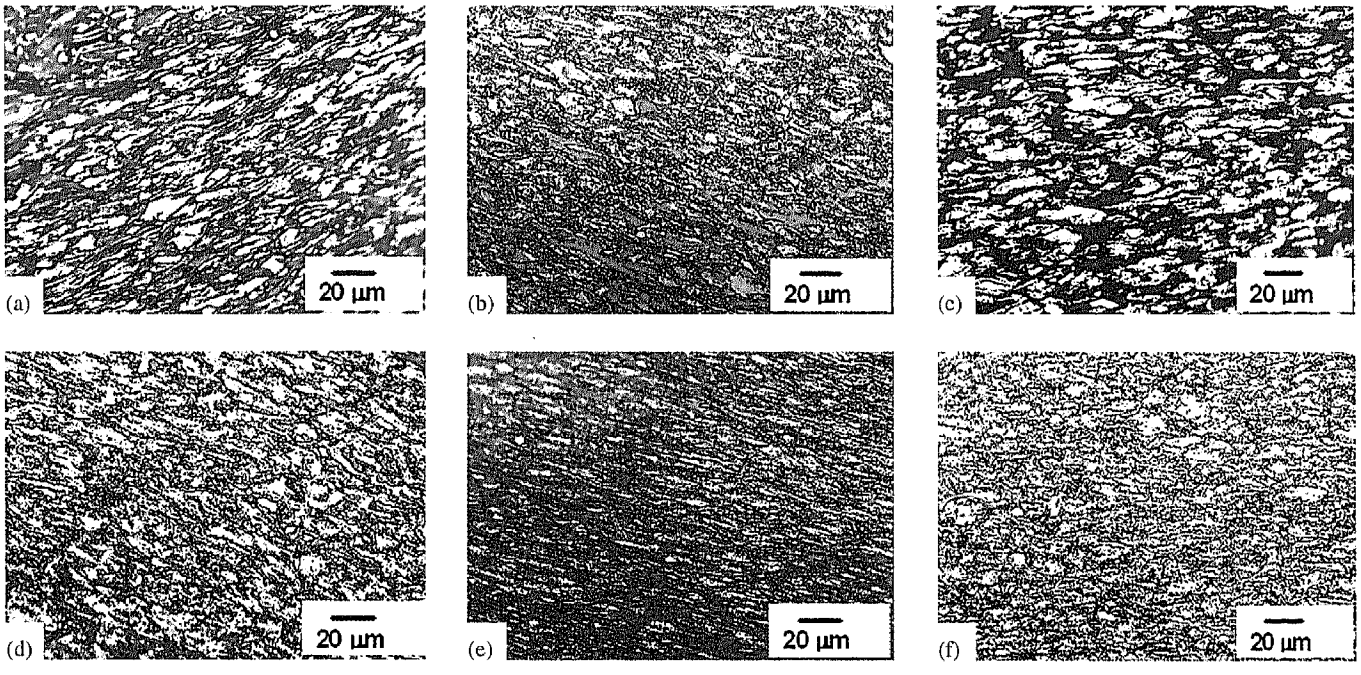

Fig. 16. Microstructure of plates rolled from THP-$325_{0.46}$ compacts before and after de-hydrogenation treatment: (a) after rolling at 200 °C; (b) after rolling at 200 °C followed by de-hydrogenation; (c) after rolling at 300 °C; (d) after rolling at 300 °C followed by de-hydrogenation; (e) after rolling at 500 °C; (f) after rolling at 500 °C followed by de-hydrogenation.

This can be explained by the three times smaller particle size and lower level of hydrogen compare to the THP-$100_{0.61}$ powder. It may be that the presence of pre-existing particle boundaries when rolling is done below 300 °C provides additional paths for hydrogen diffusion and helps to restore ductility. The results obtained at 200 °C are extremely unusual and are reproducible, however, the reason for this exceptional behaviour is not known at present and is the subject of further investigation.

3.2.7. Optical images of rolled THP-$325_{0.46}$ compacts before and after de-hydrogenation treatment

The microstructures of the rolled THP-$325_{0.46}$ only have a typical rolling structure when the temperature of rolling was 500 °C (Fig. 16). The lack of grain elongation evident in the low temperature rolling treatments may be an indication of sliding and shear at particle–particle boundaries. At the higher rolling temperature, this seems to have given way to grain elongation, as seen in the coarser particle sized powders above. Following heat treatment, the structures surprisingly look coarser than those seen for the THP-$100_{0.61}$ powders. The reason for this is not entirely clear and it may relate more to the lower H levels in this material than the finer particle size. In any event, it can be seen that the structures formed give a better combination of strength and ductility than for the coarser powders.

4. Summary and conclusions

A comparative study of the compaction of three powders THP-100, THP-325 and CP-Ti, with particle sizes 150 µm, 50 µm and 45 µm respectively, has been carried out. ECAP with back pressure was used to compact the powders. The hydride powders were compacted in the partially de-hydrogenated state.

The optimal level of residual hydrogen with respect to the density of the resulting compacts and the associated mechanical properties has been found to be 0.46 wt.% for THP-325 and 0.61 wt.% for THP-100 Fig. 17. ECAP at 300 °C produced compacts from these partially de-hydrogenated powders of 99.5 % theoretical density, while CP-Ti was compacted to almost full theoretical density under the same conditions of ECAP.

The compacts were then rolled at temperatures ranging from room temperature to 500 °C with a reduction of 80 %

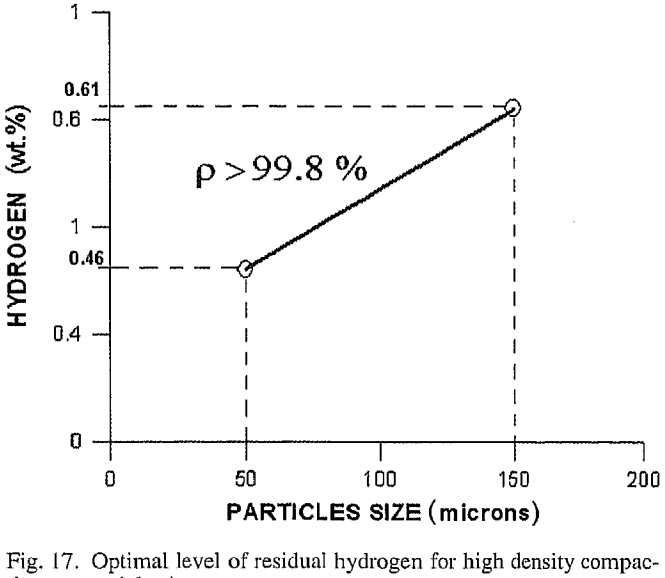

Fig. 17. Optimal level of residual hydrogen for high density compaction vs. particle size.

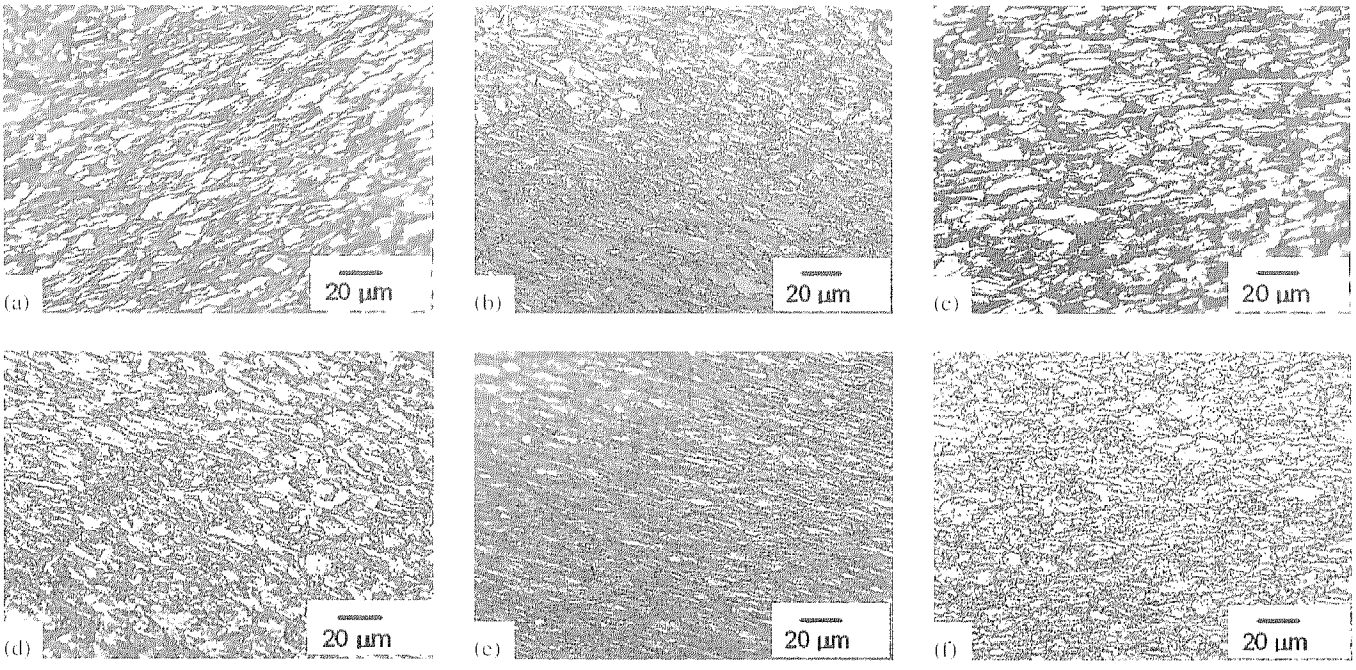

Fig. 16. Microstructure of plates rolled from THP-$325_{0.46}$ compacts before and after de-hydrogenation treatment: (a) after rolling at 200 °C; (b) after rolling at 200 °C followed by de-hydrogenation; (c) after rolling at 300 °C; (d) after rolling at 300 °C followed by de-hydrogenation; (e) after rolling at 500 °C; (f) after rolling at 500 °C followed by de-hydrogenation.

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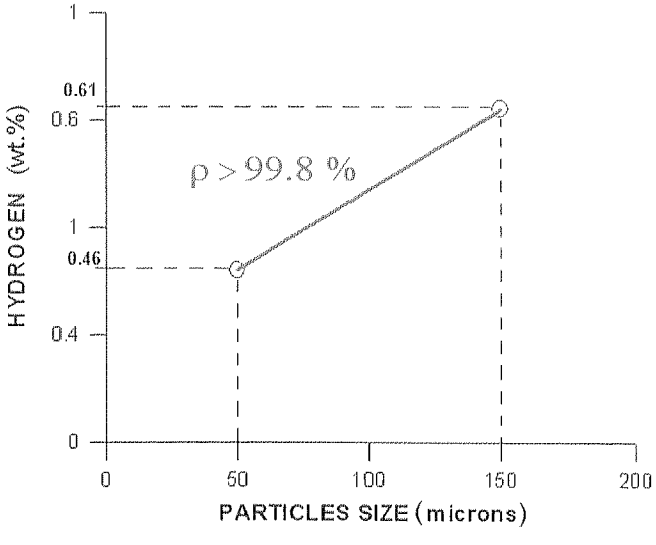

Fig. 17. Optimal level of residual hydrogen for high density compaction vs. particle size.

in a single pass. The tensile strength and ductility of samples rolled from CP-Ti compacts were compared with bulk samples rolled under the same conditions. While the strength of rolled powder compact was lower than that of the rolled bulk rod by ~45–60 MPa a slightly higher ductility of 21 % was observed.

The mechanical properties of plates rolled from partially de-hydrogenated powders were investigated by a shear punch test before and after de-hydrogenation treatment. High levels of strength and ductility were obtained, especially in the case when rolling was conducted at 500 °C. For THP-$100_{0.61}$ compact a shear punch strength of 436 MPa and a ductility of 30 % were obtained, which correspond to an equivalent tensile strength of 700 MPa and an equivalent tensile ductility of 22 %. For THP-$325_{0.46}$ compact a shear punch strength of 492 MPa and a ductility of 44.5 % were obtained, which correspond to a tensile strength of 790 MPa and a tensile ductility of 32 %. These properties significantly exceed those obtained from either the rolled CP-Ti powder compact or the plate rolled from solid CP-Ti rod.

This research shows that partially de-hydrogenated titanium hydride powders can be compacted by ECAP with back pressure and rolled at low temperatures (below 500 °C). Heat treatment after the rolling can modify the microstructure and improve the mechanical properties.

It is clear that combining ECAP with rolling is a promising route to the batch production of fully dense CP-Ti wrought products that avoids the need to subject the material to temperatures greater than 500 °C. This low temperature route is expected to be efficient from an energy point of view. It also avoids the danger of contamination that accompanies high temperature powder processing.

Compaction of CP-Ti powder using shear deformation with imposed hydrostatic pressure produces high density compacts (similar to that observed previously for Ti-6Al-4V [19]) without the addition of hydrogen and does not significantly benefit from temporary hydrogen alloying. However, the use of residual hydrogen is shown to be beneficial at the annealing/de-hydrogenation stage when the mechanical properties of the rolled compact can be improved compared to those obtained for rolled product derived from unalloyed CP-Ti powder Fig. 18. A similar improvement in the properties for ECAPed compacts through the application of a dehydrogenation heat treatment has been reported for Ti-6Al-4V powder [20].

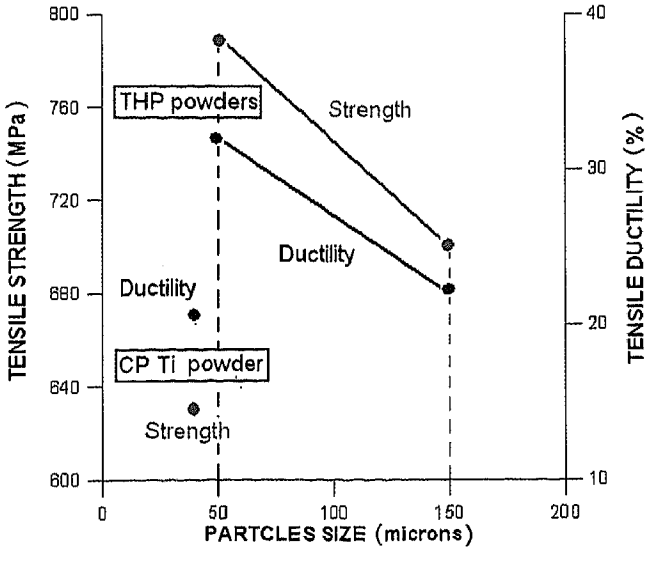

Fig. 18. Improvement in strength and ductility after dehydrogenation treatment of the rolled compacts.

This project was supported under Australian Partnership in Light Metals Research, funded as a Research Cluster under the CSIRO National Flagships Collaboration Fund. The technical assistance of Daniel Curtis is also greatly appreciated.

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(Received March 21, 2009; accepted October 14, 2009)

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DOI 10.3139/146.110226
Int. J. Mat. Res. (formerly Z. Metallkd.)
100 (2009) 12; page 1727–1738

ISSN 1862-5282

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