In situ electro-plastic treatment for thermomechanical processing of CP titanium

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Abstract
Titanium and its alloys have been used in a broad range of products such as biomedical implants due to their high specific strength, corrosion resistance, and biocompatibility. Improvement in microstructure and mechanical properties of commercially pure (CP) titanium is usually performed by cold rolling and controlled atmosphere heat treatment. However, this is an energy-intensive, expensive, and a multi-stage process to achieve the desired properties. Hence, to address these challenges, in this study, the effects of an in situ electro-plastic treatment (ISEPT) on the microstructure evolution of CP titanium have been studied. The deformation load and electric current in this treatment were applied in the same direction (in situ) to maximize the electro-plastic effect. Simultaneous electric current and strain application created a condition for dynamic recrystallization to occur at low temperature and under atmospheric conditions, thus reducing the cost and energy for manufacturing in a single process. The rapid heating and cooling prevented the oxidation of titanium to a large degree, eliminating the requirements for costly inert gas or vacuum. The results showed rapid recrystallization of CP titanium at 3.2 mm/s roller surface speed. Recrystallization led to a reduction in average grain size to 7 µm with 18% increase in microhardness. Pre-cold rolling of the CP-Ti structure enhanced grain refinement due to the introduction of dislocations and the applied electric current interactions. The SEM and XRD investigations revealed the relationship between the ISEPT treatment and the evolution of microstructure in CP-Ti. The effect of specimen geometry on the ISEPT was discussed.

Keywords Titanim · Resistant heating · Thermomechanical treatment · Electro-plastic deformation · Recrystallization · Grain refinement

1 Introduction

Commercial purity (CP) titanium material has exceptional properties of high strength-to-weight ratio, high corrosion resistance, and relatively high melting point [1]. CP-Ti is widely used in chemical processing and heat exchangers that deal with the severe corrosive environment and prosthetic parts in biomedical applications [2]. Titanium, however, is more expensive than the other rival light alloys and stainless steel partly due to the high cost of processing, such as vacuum or controlled atmosphere required during the remelting and heat treatment of ingot and downstream fabrication. The controlled atmosphere at high temperatures is necessary to prevent titanium from excessive reactivity with oxygen and hydrogen that causes embrittlement [1, 2].

The common CP titanium thermomechanical treatment (TMT) involves multi-stage hot deformation and recrystallization processes to control the size of α-phase grains and mainly convert the large lamellar microstructure to the desired fine equiaxed microstructure [1]. The starting microstructure of CP-Ti is commonly a large grain size lamellar microstructure that can reach 400 to 500 µm, which after TMT treatments can be converted to equiaxed refined grains of as fine grain size as, i.e. 10 µm. Another challenging fact is that CP-Ti has a single α-phase and non-responsive to heat treatment alone; therefore, costly and energy-consuming thermomechanical treatments are inevitable to produce the desired refined and equiaxed microstructure [1, 2]. Attempts to improve titanium’s TMT are continuous, such as the use of non-isothermal warm rolling by An et al., using a pair of rollers at a variable temperature [3]. Another work by
Zherebtsov et al. [4] on the use of room temperature gradually increasing cold rolling of CP-Ti followed by 1-hour annealing below 500 °C with consideration of twinning and dislocations evolutions during the cold working process; this work revealed three distinct microstructural stages associated with increasing deformational strain where twinning found to play important role on the microstructure evolution kinetics. These stages are (i) formation of twinning, (ii) dislocation density rise associated with formation of substructure, and (iii) formation of high angle grain boundaries and grain refinement at higher strain values [4]. Sabat et al. studied CP-Ti grade-2 hot rolling options, unidirectional rolling, multistep cross rolling, and reverse rolling to assess the texture and microstructure evolution related to the degree of % of reduction [5].

Another contemporary approach by Luo et al. was the recycling of titanium CP machining chip using equal channel angular pressing (ECAP) that reported grain refinement with improved ductility in CP-Ti [6, 7]. The use of ECAP also suggested by Yan et al., [8] to produce partially equiaxed ultra-fine grain microstructure from a near-\(\alpha\)-Ti (\(Ti\)-6Al–2Zr–1Mo–1V) alloy [8]. The two steps EACP process was carried out at a high temperature above and below \(\beta\) transus temperature followed by quenching to improve hardness. Although ECAP is considered one of the most promising severe plastic deformation (SPD) processes, it is still known to be an energy-intensive method with some technological challenges such as irregular shear straining accompanied by excessive tool and die wear [9]. In addition, the post ECAP heat treatment of CP-Ti beyond 500 °C still necessitate the expensive use of controlled atmosphere. Other studies suggested alternative near net shape manufacturing routes, including hybrid and additive manufacturing [10, 11]. The hybrid processing mainly involves powder sintering and post-consolidation treatments [12, 13], which inevitably requires a costly and energy-intensive controlled atmosphere.

The use of rapid heat treatment methods on titanium alloys has been studied for many years and showed exceptional ability in grain refinement to improve mechanical properties [14–17]. These processes include electric resistance heating, induction heating, and molten salt bath heating [14–17]. However, these works [14–17] focus mainly on annealed titanium (\(\alpha\)-\(\beta\)) alloys and not on CP-Ti grades due to its limited response to heat treatment [1]. In addition, these published works involved the use of inert gas in their process. A recent study on Ti-48Al-2Cr-2Nb by Chen et al. detailed the relationship between the current density and \(\alpha+\beta\)-phase transformation [18] without consideration of CP-Ti with \(\alpha\)-phase. Electrically assisted forming (EAF) and electric-assisted manufacturing (EAM) have revealed exceptional ability to reduce the flow stress and increase material’s formability [19, 20]. EAF involves the application of electric current during deformation that results in the “electro-plastic effect”. The electro-plastic effect is explained by three combined mechanisms of Joule heating, dislocations interaction, and metallic bond weakening that work together in reducing the deformation flow stress and increasing the material’s formability [19]. The EAF is best deployed on low formability alloys, especially titanium and magnesium alloys, with benefits.

Table 1 Properties and chemical composition of the CP-Ti strips

| Chemical composition (wt%) | Balance |
|---------------------------|---------|
| Fe                        | 0.3     |
| C                         | 0.08    |
| N                         | 0.03    |
| H                         | 0.015   |
| O                         | 0.25    |

| Mechanical properties |  |
|-----------------------|--|
| Tensile strength (MPa) | 345 |
| Yield strength (MPa)   | 275–450 |
| Elongation (%)         | 20   |
| Density (g/cm³)        | 4.7  |
to reducing deformation flow stress and spring back [19]. Xu and Chen [21] demonstrated that there is more than just Joule heating and softening effect when resistant heating was used for α-titanium deformation. The electric current was found to produce a higher heating rate introducing complicated events that benefit the EAF process. For example, the rapid heating rate caused by the momentum transfer from the electron drift into the crystal lattice improves the nucleation rate during recrystallization, which is explained by the “electron wind effect” [19, 21]. Another study [19] showed that an increase in current density while maintaining the contact temperature of 600 °C for CP titanium resulted in grain growth. The reason for this was related to an improved grain boundary migration rate induced by the electric current drift effect [19].

Salandro et al. [19] compared the effect of electro-plastic kinetics with conventional isothermal oven heating with some remarks worth considering. Firstly, it was observed that the Joule heating has different kinetics compared to isothermal heating, the former occurring by scattering electrons off interfacial defects (voids, impurities, grain boundaries) within the...
Fig. 5 LM microstructures of CP-Ti wire cross-section, a, b before ISEPT, c, d at 0.52 mm/s, e, f at 3.19 mm/s, and g, h at 6.4 mm/s RSS
lattice, while the latter occurring by vibrational energy transfer via convection or radiation that results in uniform temperature distribution. Resistance heating is nonuniform, and it results in a higher temperature in the region surrounding the interfacial defects with higher energy. It is caused by electrons scattering, compared to other regions, although the gained vibrational energy will spread to the other regions forming temperature gradients. Secondly, direct dislocation-electron interaction assists dislocation motion in the case of electric resistance heating. Thirdly, the electron flow plays an additional role in weakening the metallic bond, resulting in further softening and flow stress reduction [19]. The use of continuous electric resistance heating followed by plastic deformation was studied by Yanagimoto and Izumi for low formability metals such as titanium and stainless steel [22]. In this work, the EAF and electro-plastic effect were applied in two separate stages. Another work by Saifullin and Natalenko proposed electric resistance heating of sintered sheets using a steel mesh to accommodate the powder for strip manufacturing [23].

The electric-assisted compression test revealed that temperature declines during deformation, indicating the supplied power was partially consumed in assisting dislocation motion [19]. The electric-assisted deformation’s stress-strain profile also showed a drop in flow stress and prolonged formability compared to the isothermal treatment [19]. The resulting microstructure after EAF processing of titanium consisted of equiaxed grains with variable grain size, a character of dynamic recrystallization caused by electric resistance heating. Salandro et al. [24] studied the temperature variation through the cross-section that led to a change in heat input and straining that affected the nucleation rate and grain growth. Comparing the stress-strain profile of the EAF test to the schematic presentation of the flow curves for dynamic recovery and dynamic recrystallization, published by Zahiri et al. [25], also showed the similarity of the dynamic recrystallization curve with the presence of peak stress followed by a decrease in flow stress.

Titanium reacts with oxygen at approximately 550 °C [1]. Previous work by An et al. [3] revealed the ability to completely recrystallize cold-worked CP-Ti at 450 °C using non-isothermal warm rolling with a 90 °C temperature gap between the two rollers. This indicated the possibility for recrystallization to occur at temperatures below the critical temperature for the oxidation of titanium (500–600 °C) [1], where the internal energy works as the driving force for dynamic recrystallization. Humphrey et al. [26] showed that the recrystallization temperature decreases as the thermomechanical process strain increases. A low heating rate encourages dynamic recovery reducing the required driving force for recrystallization [26].

This study aims to investigate the effect of in situ electro-plastic treatment (ISEPT) on CP-Ti recrystallization and grain refinement. It was demonstrated that under certain conditions, simultaneous application of current and load in ISEPT results in partial and fully recrystallized material. The effect of deformation speed (strain rate) and sample geometry on the formation of new grains were discussed.

2 Materials and methods

2.1 In situ electro-plastic treatment

The ISEPT process used in this study consisted of a machine that was developed at the Commonwealth Scientific and Industrial Research Organization (CSIRO) under patent no WO2018232451 [27]. The schematic diagram in Fig. 1 shows that machine’s two rollers were connected to the AC power source. The specimen was fed to the machine between two rollers via lifting the top roller using a piston and interlocked back to connect to the electric circuit through the specimen. The current was applied in the same direction as the applied force to simultaneously heat and deform the specimen.
2.2 Materials used

Three types of commercially pure titanium (CP-Ti) specimens were used to assess the effect of electro-plastic treatment on recrystallization with consideration of the sample (cross-section) geometry:

(a) CP-Ti wire: Readymade annealed wire of 2.38 mm diameter
(b) CP-Ti strips: Two strips of machined rectangular cross-sections 6.5×5 mm and 5×3 mm
(c) Cold-worked CP-Ti strip: Rectangular cross-section 3×5 mm and cold-rolled to 30% reduction in thickness

The rectangular strips were machined from a CP-Ti grade-2 plate with composition and properties shown in Table 1.

The microstructure of the CP-Ti wire and CP-Ti strips was examined before and after the electro-plastic treatments. Samples were cut perpendicular to the rolling direction in all experiments and cold mounted in epoxy moulds, grounded progressively down to 2000 grit using SiC papers for light microscopy. Overheating during grounding and cutting was avoided using a cooling medium. Progressive polishing down to 1 μm was used, followed by the final OP-S mix with ammonia and hydrogen peroxide etch-polishing.

The mechanical property of CP-Ti samples before and after electro-plastic treatment was measured by HV microhardness under 300-N load to quantify materials softening behaviour. LECO Inert gas Fusion Analyzer Model-ONH836 series, by LECO Corporation, USA, was used to determine oxygen and nitrogen content before and after treatments. All electro-plastic experiments were conducted under atmospheric conditions in the absence of inert gas. SEM imaging was carried out using a ZEISS, Merlin GeminiSEM series, Oberkochen, Germany, electron microscope. A Bruker D8 Advance A25 XRD machine, Bruker Corporation, USA, was used to reveal CP-Ti samples X-ray peaks.

3 Results

The main process parameters for the electro-plastic treatment of this study were the applied load, applied current, and the rolling surface speed (RSS). The effect of these parameters on CP-Ti microstructure, grain size, and microhardness was determined.

![Fig. 8 LM image of CP-Ti strip](image)

(a)

(b)

![Fig. 9 Grain boundaries serration as reported by Humphreys et al.](image)

![Fig. 10 Effect of RSS on thickness reduction for 5×3 mm cross-section CP-Ti strip at 1.5 kA and 0.7 MPa](image)
3.1 Electro-plastic treatment of CP-Ti wire

A stock of annealed CP-Ti wire of 2.38 mm diameter was used in the ISEP treatment. The applied 0.7 MPa pressure and 1.5 kA current were kept constant in all experiments, and the RSS was gradually increased from 0.52 mm/s (0.038 s\(^{-1}\) strain rate) to 19.5 mm/s (0.74 s\(^{-1}\)), as shown in Fig. 2. The results showed an increase in RSS led to the rise in the strain rate with a less achievable reduction in thickness. At the lowest RSS of 0.52 mm/s, the sample thickness reduced by 58.6%. This was equal to 0.88 true strain at a strain rate of 0.04 s\(^{-1}\) compared to the highest RSS of 19.5 mm/s, which resulted in a 0.3 true strain and 0.74 s\(^{-1}\) strain rate with a 26% reduction.

Figure 3 revealed that, overall, an increase in rolling speed above 0.52 mm/s resulted in an improvement of hardness for conditions of this study. The hardness and grain size of the electro-plastically deformed materials were less than the original wire. Finer grains were created with an increasing strain rate up to RSS of 3.19 mm/s (0.18 s\(^{-1}\) strain rate), which was in agreement with the observations in the published literature [26]. However, a further increase in RSS (strain rate) above 3.19 mm/s retarded recrystallization with the formation of elongated grains. The samples showed insignificant change in oxygen and nitrogen content before and after ISEPT, as shown in Fig. 4. This is believed to result from the rapid heating rate and the lower recrystallization temperature associated with this treatment.

Figure 5 shows light microscope (LM) images of the CP-Ti wire cross-section before and after the treatment. The wire’s initial microstructure had an average grain size of 25 \(\mu\)m, as seen in Fig. 5 a and b. Recrystallization and the X pattern of grain refinement were caused by strain variation along the cross-section, as shown in Fig. 5 c, e, and g which was in agreement with previous studies [28]. A minimum reduction of 35% was required to observe new dynamically recrystallized grains in the microstructure. Results in Figs. 3 and 5 suggested that an increase in RSS led to a reduction in dynamic recrystallization for conditions of this study. It is worth noting that for simplicity, we use the term “dynamic recrystallization” to include meta-dynamic recrystallization [29, 30] as further study is required to differentiate these two processes under ISEPT conditions.

3.2 Electro-plastic treatment of CP-Ti strips

To determine the effect of sample geometry and size on ISEPT, CP-Ti strips with 6.5×5 mm (32.5 mm\(^2\)) and 5×3 mm (15 mm\(^2\)) with rectangular cross-section were prepared, and the cross-section dimensions are \(w_0\) and \(h_0\) (Fig.
The initial microstructure for the CP-Ti strip had an average grain size of 68 μm (Fig. 6).

The 6.5×5 mm CP-Ti strips were treated under similar processing conditions as the wire with a constant 1.5 kA current and 0.7 MPa pressure to compare the effect of RSS on (dynamic) recrystallization. As represented in Fig. 7, the results show a maximum 17.8% reduction in thickness at the lowest surface speed of 0.52 mm/s. Under this condition, a microstructure with grain boundary serration (Fig. 8a) was observed that resembled the first stage of dynamic recrystallization, as shown in Fig. 9 sourced from [26].

Recrystallized grains were not evident in other 6.5×5 mm (32.5 mm²) samples when higher RSS than 0.52 mm/s was used. Insufficient (11%) reduction caused a lamellar microstructure with laths and twins at 1.16 mm/s surface speed, as shown in Fig. 8b. Further increase in rolling speed contributed to a 7.7% reduction (Fig. 7) and less apparent microstructure changes.

### 3.3 Effect of the strips’ cross-section

The effect of geometry on the CP-Ti ISEPT was studied using the rectangular samples with a 5×3 mm cross-section that was ~50% less area than the previous 6.5×5 mm samples, which were deformed under two orientations. In the first orientation, the aspect ratio (AR), that is, the ratio of width to height of the strip, was considered to be <1. Therefore, w₀ in Fig. 1b was 3 mm, and h₀ was 5 mm when the 5×3 strip was deformed. The results showed an increase in the rollers’ surface speed led to less deformation and straining of the samples (Fig. 10). The largest value for the reduction was ~34% at the lowest RSS of 0.52 mm/s. Under the conditions of this study, higher rolling speeds above 2.5 mm/s did not contribute to further reduction indicating the upper bound of rolling speed was reached.

The LM images corresponding to deformed samples in Fig. 10 are presented in Fig. 11. Compared with Fig. 6, partial recrystallization and necklacing were initiated at 0.52 mm/s and 1.16 mm/s rolling speeds, as shown in Fig. 11a. Further increase in rolling speed above 1.16 mm/s (Fig. 11b, c) did not provide sufficient driving force for detectable recrystallization, mainly leading to the formation of twins and grain boundary serration. This was similar to the conditions for cold rolling of CP-Ti in earlier studies [31, 32] that demonstrated the dominant mechanism for deformation at low straining, i.e. deformation below 40% is mainly slippage and twinning.

The effect of sample contact surface on the rollers under identical ISEPT conditions can be verified from Figs. 7 and 10. The sample with smaller 3 mm width and AR <1 had the highest amount of deformation (34.2%). The contact surface difference between the 3- and 6.5-mm-wide samples was at its maximum (~50%) when the lowest RSS was applied.

### 3.4 Effect of the applied current

Figure 12 illustrated the influence of current on the electroplastic deformation of 5×3 mm CP-Ti strips when AR was <1. An increase in the applied current resulted in a higher reduction in thickness. The LM images related to Fig. 12 are shown in Fig. 11, Fig. 13, and Fig. 14. The results for the 2kA samples were mixed with the creation of refined recrystallized grains in some areas at 0.52 mm/s RSS and the formation of twins that became highly refined at the fastest 3.19 (mm/s) RSS (Fig. 13a–c). A similar trend in the evolution of microstructure was observed at 2.5 kA (Fig. 14a–c) that require further studies to determine the critical stress for the initiation of dynamic recrystallization.

Results in Fig. 12 suggest that at least 34% deformation was required to initiate recrystallization in CP-Ti for conditions of this study. Overall, recrystallization at a lower current and slower surface speed (i.e., 1.5 kA and 0.52 mm/s) was more favourable for the initiation of dynamic recrystallization, as observed in Figs. 11, 13, and 14. Furthermore, the benefit of using less current in ISEPT manufacturing was to reduce energy consumption and to prevent excessive heating that could cause oxygen and nitrogen reaction with CP-Ti under atmospheric conditions.

Qualitative comparison of the microstructures in Figs. 13a and 14a showed the appearance of more recrystallized grains when current increased from 2 to 2.5 kA. In agreement with an
Fig. 13  LM images of 3×5 mm CP-Ti strip microstructure after ISEPT at 2.0 kA and 0.7 MPa corresponding to a 0.52 mm/s, b 1.85 mm/s, and c 3.19 mm/s RSS.

Fig. 14  LM images of 3×5 mm CP-Ti strip microstructure after ISEPT at 2.5 kA and 0.7 MPa corresponding to a 0.52 mm/s, b 1.85 mm/s, and c 3.19 mm/s RSS.
earlier study [19], a nonuniform mixture of large and small grains, as shown in Fig. 14a, was observed at the highest applied current. Figures 11b–c, 13b–c, and 14b–c show further coarsening of a lamellar structure with twins in the absence of recrystallization; this could be explained by the retained heat in the samples at higher rolling speed. A similar event occurs in hot rolling processes that are caused by a short time of contact between rollers and material at high rolling speeds [33].

3.5 Effect of rolling orientation

To study the effect of specimen rolling orientation on the in situ electro-plastic deformations, the 5×3 mm sample was rotated 90° and rolled in such a way that the 5 mm side of the sample faced the rollers surface denoted as “horizontal orientation”. This corresponded to \( w_0 = 5 \text{mm} \) and initial roller gap \( h_0 = 3 \text{mm} \) in Fig. 1b with AR of the strip >1. This study was to compare the results with “normal orientation” conditions when strip AR was <1. Compared with normal orientation, a significant decrease in reduction from 34 to 18% was observed after horizontal deformation (Fig. 15). The electro-plastic treatment’s sensitivity to the geometry and rolling orientation was further highlighted by the LM images in Fig. 16, which shows the absence of detectable recrystallized grains in horizontal specimens compared with recrystallized CP-Ti in Fig. 11a in normal orientation. This suggested that under identical conditions, ISEPT samples with AR<1 exhibit improved softening and recrystallization. Figure 16b and c revealed formation of twins at higher RSS that led to a short time of exposure to current and reduced heating of the strip. This led to less reduction ~18% and a similar condition for cold-rolled CP-Ti reported by Chun et al. [32].

3.6 Effect of cold working

To examine the role of prior cold working on the effectiveness of the ISEPT, a CP-Ti strip (5.25×3 mm cross-section) was cold-rolled in the normal direction to 30% of its original thickness. The strips were rotated 90° to the direction of cold-rolled material to achieve aspect ratio smaller than 1. The ISEPT conditions were 1.5 kA and 0.7 MPa with similar RSS to previous experiments. Figure 17a shows grain refinement at all RSS compared with the previous experiments without cold working, and the least achieved % reduction in thickness was ~28% at RSS of 3.2 mm/s. The microhardness of the stock, 30% cold-rolled, and post ISEPT treatment of the strips are shown in Fig. 17b. Results show that cold rolling increased CP-Ti hardness from HV-145 to HV-215 due to strain hardening, followed by a reduction to HV-172 after ISEPT recrystallization. The effect of RSS on microhardness was marginal as shown in Fig. 17b. Figure 18 presents the pre-cold-worked and electro-plastic treated microstructure at 0.52 mm/s resulted in a mainly uniform recrystallized microstructure with finer grain size than the original strip shown in Fig. 6.

A comparison of the original strip microstructure with 30% cold-rolled and ISEPT-treated material are shown in Fig. 19a–f that were in agreement with the literature [26] on the effect of prior cold working on providing the required driving force for recrystallization. These qualitative results highlighted the contribution of electron flow interactions with dislocations and crystallographic defects in providing sufficient Joule heating and dislocation motion assistance to improve microstructural refinement [19]. Comparing the images in Fig. 19b, d, and f, we can observe the presence of some twins in ISEPT CP-Ti in Fig. 19f was most likely due to the rapid heating nature of ISEPT compared to the conventional slow isothermal heating process that generally results in a twin free microstructure.

The microstructure of the cold-rolled and electro-plastic treated specimens at 0.52 mm/s RSS is shown in Fig. 19e–f knowing that the initial grain size of the untreated strip was 67 \( \mu \text{m} \), an increase in rolling speed resulted in marginal grain refinement and microhardness values. For example, at the two RSS extremes of 0.52 and 3.2 mm/s, an average grain size of 12 \( \mu \text{m} \) and 8 \( \mu \text{m} \) was corresponded to microhardness values of HV-170 and HV-172, respectively. These results indicated the capability of the in situ electro-plastic deformation to considerably refine CP-Ti grain in a relatively short process time and without the need for inert gas or vacuum.
Further to this, Fig. 19 e–f and Fig. 17 a confirm the ability of ISEPT to obtain finer grains with increasing strain rate in agreement with the literature [26]. A potential benefit of this is to manufacture ultrafine grain structures (i.e. grain size \(<5 \mu m\) [25] to further improve the mechanical properties of CP-Ti. That could be the subject of another study.

4 Discussion

Practical expressions that explain the relationship between the ISEPT parameters and developed microstructure generally consider the deformation variables such as the amount of reduction, strain, and strain rate in addition to current density and Joule heating.

Equation (1) estimates the percentage of the reduction [34]:

\[
\% \text{reduction} = \frac{h_0 - h_f}{h_0} \times 100
\]

where \(h_0, h_f\) is the sample thickness before and after deformation, respectively (Fig. 1b).

The relationship between the applied strain and strain rate is expressed as Eq. (2).

\[
\dot{\varepsilon} = \frac{RSS}{L_c} \left( \varepsilon \right) = \frac{RSS}{L_c} \left( \frac{\ln \frac{h_0}{h_f}}{C_18/C_19} \right)
\]

where \(\varepsilon\) is the true strain, \(\dot{\varepsilon}\) is strain rate, \(RSS\) is the rollers rotational surface speed, and \(L_c\) is the contact length between the rollers and sample (Fig. 1b) estimated from Eq. (3).

\[
L_c = \sqrt{r(h_0-h_f)}
\]

where \(r\) is the roller’s radius.

The rolling time \((t)\) in Eq. (4) is derived from Eq. (2). The current application time (CAT) or heating time was considered similar to the rolling time for ISEPT due to the application of load and current under the same direction and simultaneously (Fig. 1b).

\[
t = \frac{L_c}{RSS}
\]

The current density \(J\) was estimated using Eq. (5).

\[
J = \frac{I}{A}
\]
where $I$ is the applied electric current and $A$ is the contact area ($L_c \times w_f$ in Fig. 1b), the area normal to the applied current direction [19, 35].

Equation (6) approximates the Joule heating energy:

$$Q = I^2 R t$$  \hspace{1cm} (6)

where $I$ is the applied current, $R$ is electrical resistance, and $t$ is CAT estimated from Eq. (4) [35].

The samples resistance $R$ in Eq. (6) is determined from the material’s electrical resistivity and the sample dimensions (Fig. 1b) as Eq. (7):

$$R = \frac{\rho h_f}{A}$$  \hspace{1cm} (7)

where $\rho$ is the material electrical resistivity (estimated for CP-Ti = 0.000052 $\Omega$-cm), $h_f$ is the rollers gap in this study, and $A$ the contact area ($L_c \times w_f$) in Fig. 1b.
Fig. 19  LM and SEM images of CP-Ti strip a, b as received microstructure; b, c the microstructure after 30% cold working; e, f recrystallized after ISEPT at 1.5 kA, 0.7 MPa, and 0.52 mm/s RSS

Fig. 20  The current density and Joule heating energy as a function of RSS for in situ electro-plastic processed CP-Ti wire at 1.5 kA and 0.7 MPa
4.1 Effect of rolling surface speed (strain rate)

Figures 20 and 21 show the estimated current density (Eq. (5)) and Joule heating (Eq. (6)) of the ISEPT in relation to applied RSS. The lowest RSS (0.52 mm/s) led to the lowest current density and the highest Joule heating that coincided with the formation of CP-Ti recrystallized grains (Fig. 11a). In contrast, high RSS, i.e. 3.19 mm/s (Fig. 20), caused the maximum current density and minimum Joule heating, resulting in excessive heating [33] from the short contact time and more effective roller chill.

Equation (7) shows a more pronounced effect of RSS at lower speeds on the CAT and reduced material resistance (Fig. 21) that raised the Joule heating energy in Fig. 20. The extended exposure of the sample at lower rolling speeds, i.e. 0.52 mm/s, was associated with the highest 22.86 s CAT that was related to the largest deformation (58%) in Fig. 2.

4.2 Effect of sample geometry and aspect ratio

Figure 22 shows the results for the effect of sample cross-section on current density and Joule heating. For example, at 0.52 (mm/s) RSS, the CP-Ti wire, due to its circular and smaller cross-section, showed the highest value, 29.7 A/mm² (Fig. 23a). In contrast, the current density for 6.5×5 mm strip at 0.52 mm/s RSS was the lowest (21.9 A/mm²) with...
increased contact area as expected. The highest current density at the fastest RSS (3.19 mm/s) had a limited effect on 6.5×5 mm strip deformation with the lowest 4.3% reduction in Fig. 7. This was the consequence of a considerable decrease in current application time and Joule heating, as shown in Fig. 22b.

Figure 22b shows that the estimated Joule heating energy from Eq. (6) increased with an increase in the sample’s cross-section. In contrast to the wire with 58.6% reduction, the 6.5×5 mm strip with 1.9 kJ did not achieve sufficient straining (17.8% reduction) for recrystallization, as shown in Fig. 8a. The consequence of this was mainly grain boundary serration. The 5×3 mm strip with 1.55 kJ and 22.8 A/mm² deformed 34% with improved (partial) recrystallization, as seen in Fig. 11a. It is worth noting that all conditions that allowed for some degree of softening prevented CP-Ti under atmospheric conditions to react with oxygen and nitrogen, as shown in Fig. 4 chemical analysis. In this regard, the ISEPT of CP-Ti reduced the temperature and exposure time for the material to a level that the controlled atmosphere was unnecessary. From the samples’ microstructure in Figs. 5, 8, 11, 13, and 14, it is confirmed that excessive Joule heating does not improve the recrystallization, an outcome that was in agreement with the literature [19, 26].

The effect of strip aspect ratio (the ratio of wₒ to hₒ) on current density and Joule heating is presented in Fig. 23. An
increase in RSS led to an increase in current density (Fig. 23a) with the opposite effect on Joule heating (Fig. 23b) that was consistent with earlier estimations for ISEPT of CP-Ti wire. The most considerable reduction and recrystallization of the 5×3 mm strip were observed (Fig. 14a) when a combination of the lowest current density and the highest Joule heating achieved for the AR<1. These outcomes could be utilized to optimize ISEPT in relation to saving energy for the manufacture of CP-Ti mill products.

A more detailed analysis of the ISEPT parameters for the 5×3 strip at RSS 0.52 mm/s is shown in Fig. 24. The highest (34%) reduction was achieved when the sample had a normal orientation (AR<1). The resistance of the two samples had only a 10% difference. In contrast, CAT was 26s, almost double under normal rolling direction due to the larger contact length. The horizontal (AR>1) rolling resulted in greater current density but much lower Joule heating energy values, and the latter is believed to be the reason for the low softening effect and reduced straining. The LM images of the horizontal rolling shown in Fig. 16 revealed the absence of recrystallization and grain refinement because of the limited 18% deformation. These results revealed an increase in current density without optimization of Joule heating energy has a limited effect on the initiation of recrystallization.

4.3 Effect of pre-cold rolling

Recrystallized microstructures in relation to all RSS were only achieved when the CP-Ti strip was cold-rolled prior to the ISEPT (Fig. 19). The effect of pre-cold work was to introduce dislocations that act as obstacles to the electron drift through the materials [19]. Furthermore, a higher number of dislocations in the material increased the nucleation sites for new grains [26]. Another benefit of the dislocations that differentiated the current electro-plastic treatment with, i.e. warm rolling, was the interaction of the electric current (electrons) with the high-density dislocations that have been suggested to accelerate recrystallization [19]. Further studies that include measurement of the changes in CP-Ti resistivity via cold working will be beneficial to understand its effect on ISEPT parameters.

A comparison of the normalized Joule heating energy for all samples in Fig. 25 allowed for comparison of the results in relation to applied RSS. Results for the pre-cold-worked samples were not included due to insufficient information about the changes in resistivity of the CP-Ti under deformation conditions of this study. The 2.38 mm Dia wire and the 5×3 normal rolling orientation samples had matching plots at RSS 0.52 mm/s and 1.16 mm/s with 30 to 42 A/mm² current density. The lowest values for normalized Joule heating were associated with 5×3 mm horizontal rolling orientation highlighted the importance of sample contact area on the ISEPT effectiveness for recrystallization.

Further analysis of the ISEPT on the CP-Ti was carried out using XRD (Fig. 26). The XRD phase analysis presented only hexagonal α-titanium phase. Higher texture was observed in the cold-rolled (CW) and ISEPT-treated samples, while a small amount of texture at the same direction was observed in the stock and post ISEPT cold-rolled (CW + ISEPT) samples. This was evident by the relative intensity of the (002) peaks and other peaks with L-index of four specimens. The texture analysis revealed that cold rolling followed by 90° rotate ISEPT eliminated the deformation texture resulting in a microstructure comparable to the stock annealed CP-Ti.

To clarify the effect of ISEPT on the lattice parameters, crystallite size, and the microstraining, the stock CP-Ti strip was compared with three chosen strips that were 30% cold...
worked, ISEPT processed, and 30% cold-worked followed by ISEPT as shown in Fig. 27a and b. The strain of the cold-rolled strip showed the expected rise as presented in Fig. 27a. The annealed CP-Ti strip showed the largest crystallite size as expected due to the slow isothermal heating commonly used in the commercial TMT. Compared to the stock material, XRD results confirmed the SEM results in Fig. 19 that showed ISEPT refined CP-Ti microstructure. According to a previously published work by Jandaghi et al. [36, 37], the interplanar atomic spacing (d-spacing) has a direct relation to the material’s electrical conductivity, where small or narrow interplanar spacing causes higher electrons collision with the ions cores due to the limited space for movement of the drifted electrons in the crystal lattice. Jandaghi et al. [37] also reported the important role of plastic deformation and severe straining in “pulling the trigger” of dynamic recrystallization. In their work, crystal lattice swelling after cold rolling corresponded to the increase in d-spacing. This lattice swelling was explained by dynamic inhalation of dislocation via dynamic recrystallization [37]. A comparison of the d-spacing calculations for four selected samples in Fig. 27b showed insignificant variation in the interplanar spacing in both a and c parameters with slight increase in the cold-rolled sample parameters. This increase could be resulted from the lattice swell reported by Jandaghi et al. that requires future investigation.

5 Conclusions

The effect of key process parameters of an in situ electroplastic treatment (ISEPT) on microstructure, grain size, and microhardness of an annealed and cold-worked CP-Ti was investigated. The ISEPT of CP-Ti was conducted in air and without a controlled atmosphere. The undetectable variations in oxygen and nitrogen content before and after the ISEPT suggested that recrystallization temperature was lower than the temperature for the oxidation of titanium. In this study, the process parameters were found to be highly dependent on the initial microstructure of the material. Introduction of higher dislocation density sourced from cold deformation improved recrystallization most likely due to the interaction of the material with the electrons flow. The ISEPT considerably reduced the texture corresponding to the cold-worked CP-Ti with limited effect on lattice parameters.

The optimum ISEPT parameters to initiate the formation of new grains were quantified as a minimum of 35% deformation with a maximum 3.19 mm/s surface speed at 1.5 kA...
equivalent to 42 A/mm² current density. The rolling surface speed (strain rate) affected current density, Joule heating energy, and specimen’s achievable deformation.

It was found that the ISEPT led to improved recrystallization when the specimen rolling aspect ratio was <1. The rapid nature and low energy consumption of the ISEPT seem to be advantageous in relation to the conventional thermomechanical treatments to produce titanium mill products allowing utilization of this material in broader engineering applications.

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Syed H. Masood: Primary supervisor of this project, working with MAK, and contribution towards manuscript
Suresh Palanisamy: Co-supervisor of MAK, contribution towards materials characterization, and writing of the manuscript
Stefan Gulizia: Co-supervision

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Data availability The raw/processed data and materials required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

Declarations

Ethical approval There are no requirements for ethical approval for this project.

Consent to participate/publish All the authors consent to participate and contributed towards this work and publication in IJAMT journal.

Conflict of interest The authors declare no competing interests.

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