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Formation of Deformation Twin and Sub Gain for Strengthening of CP-Ti through RCSR with Multiple Number of Passes

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This research looks at the microstructure and mechanical properties of commercially pure titanium that has been subjected to the Repetitive Corrugation and Straightening by Rolling (RCSR) and Intermediate Annealing processes. The RCSR methodology offers notable advantages, including process simplicity, operation at room temperature, and seamless integration with existing industrial rolling systems. Unlike other severe plastic deformation (SPD) techniques that require complex and specialized tooling, RCSR stands out as a more practical and industry-friendly approach.Scanning Electron Microscope was used to analyse the microstructure of deformed specimens that had multiple passes. The image revealed that deformation twining and sub grain formation were observed after RCSR, and that this increased as the number of passes increased.The intermediate annealing allowed the number of passes to be increased from 10 to 14. Vickers hardness tester was used to determine the microhardness. Due to the dynamic recrystallization (DRX) process, the microhardness was increased by 87 percent after 14 passes when compared to base metal without RCSR. After 14 passes,the specimen has become stronger and has formed a surface crack.This comprehensive study uniquely investigates the microstructural evolution and hardness properties of CP-Ti subjected to up to 14 passes of RCSR at room temperature, followed by an immediate annealing process.

Keywords: Repetitive corrugation and straightening by rolling; deformation twin; microhardness; intermediate annealing

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Nano crystalline materials have become more common in industry due to their superior physical, mechanical, and tribological properties when compared to traditional coarse-grained polycrystalline materials. Nanocrystalline materials can be prepared in several ways such as solid state processing (Mechanical alloying [1–3], Severe Plastic Deformation (SPD) [4–7], spark plasma sintering [8, 9]), liquid processing (melt spinning[10], rapid solidification [11, 12]), Vapor-phase processing (Metalorganic vapor-phase epitaxy (MOVPE), sputtering [13]), and solution processing (electrodeposition [14]). Of all these techniques, SPD is used for processing of bulk ultrafine materials without expense of materials. Severe plastic deformation is one of the novel metal forming techniques that involves very large amount strain to achieve nanocrystalline materials. It also entails applying a complex stress condition or high shear, resulting in nanocrystalline structure (less than 100 nanometers) and ultrafine grains (100-500 nm). There are different SPD techniques available such as Equal Channel Angular Pressing (ECAP) [15–17], Constrained Groove Pressing (CGP) [18], High Pressure Torsion (HPT) [19, 20], Cyclic Extrusion and Compression (CEC) [21], Accumulative Roll Bonding (ARB) [22, 23], Multidirectional Forging (MF) [5], Twist Extrusion (TE) [24], Repetitive Corrugation and Straightening) (RCS) [25, 26] . ECAP, CGP, HPT, and RCS are the most effective SPD techniques, based on the aforementioned techniques. ECAP is a technique for extracting nano grain size from coarse grain using the SPD technique. It is, however, only suitable for batch processing and is difficult to scale up for use in a number of industries. When opposed to ECAP, CGP develops a lot of plastic strain [18]. The plastic strain distributions, on the other hand, are not uniform, resulting in heterogeneous benefit formation. The combination of compression and torsion of the specimen allows HPT to create nano grain structure. It is, however, only applicable to specimens of small size, such as those with a maximum disc diameter of 20 mm and a thickness of 1 mm. The most popular SPD techniques for obtaining bulk nanocrystalline structure are RCSR or RCS. RCS has the benefit of providing contamination-free and porosity-free materials. It's also ideal for industrial development

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on a wide scale. Sheet material is processed via a corrugated and straightening roller in RCSR, causing the material to be subjected to a considerable amount of plastic strain. This results in grain refinement. RCS study usually focused on various alloys such as aluminum, steel, copper and magnesium. Titanium is a potential material for many industrial applications due to its high tensile strength to density ratio, high corrosion resistance, fatigue strength, and ability to withstand high temperatures. In ECAP and cold extrusion, the transverse microhardness of Commercially Pure Titanium (CP-Ti) billet was increased by 6.67% with a 47 percent reduction in specimen. After a 75% reduction of the specimen by cold extrusion, the microhardness increased by 19.62%. The longitudinal microhardness of ECAP and cold extrusion was increased by 4.6% and 19.2%, respectively, with a decrease of 47% and 75% [27]. In the longitudinal direction, the microhardness value increased 163 ± 5.52 to 232 ± 5.33, while in the transverse direction, it increased 146 ± 2.13 to 220 ± 15.09. The highest value was obtained in the longitudinal direction as stated in [17]. There are two types of mechanisms that lead to ultrafine grain structure: elongated lamellar boundary structure along Rolling Direction and equiaxed grain. The CP-Ti reported by Daisuke Terada et al. was processed by ARB. Groove Pressing (GP) and CGP are two basic shear deformation processes for grain refinement. As a result, the microhardness of GP-processed materials increased from 190 after 4 passes to 203 after 8 passes, while CGP-processed materials increased from 208 to 209 after 8 passes at 300°C. Due to strain reversal during successive passes by dislocation annihilation mechanism, the microhardness value decreased to 183 after 3 GP passes from 203 after 2 passes, as stated [28]. Hydrostatic extrusion (HE) was employed to enhance the machinability and mechanical properties of Ti Grade 2 titanium. The researchers achieved a true strain value of 2.28, resulting in a remarkable 190% improvement in the Ultimate Tensile Strength (UTS) following the HE process [29]. Further studies revealed that the combination of Equal Channel Angular Pressing and Rotary Swaging (ECAP/RS) enhanced the average yield strength to 1383 MPa while maintaining a moderate ductility of approximately 10%. In addition to improved mechanical properties, the fatigue endurance limit increased by 43%, reaching up to 600 MPa [30]. Moreover, SPD techniques are emerging as highly effective methods for grain refinement by introducing extremely high levels of plastic deformation, leading to significant accumulation of total plastic strain [31]. The majority of studies in the literature focused on using ECAP, ARB, CGP, and HPT techniques to improve the mechanical properties of CP-Ti. Just a few works on RCSR on titanium with a higher number of passes were available. However, no comprehensive research has been conducted on the microstructural evaluation and hardness characteristics of CP-Ti processed via RCSR up to 14 passes. This study would be the first of its kind to explore the technique at such a high level of deformation intensity. The aim of this research is to look into the microstructure of CP-Ti that has been processed by RCSR with a high number of passes, up to 14, in order to improve mechanical properties like hardness level without compromising formability. This investigation would be extremely beneficial in the development of a component for aerospace and biomedical applications that necessitates increased strength and hardness without modifying chemical compositions. Methodologically, RCSR provides process simplicity, usability at room temperature, and compatibility with industrial rolling systems, in contrast to other SPD techniques that necessitate specialized tooling. Nonetheless, constraints like as strain homogeneity across thickness and possible anisotropy in grain refinement require systematic attention, particularly at elevated pass counts. The expected outcomes such as boosted surface hardness and strength due to grain refinement, Analysis of microstructural patterns of strain buildup, Evaluation of the potential of RCSR as an industrially scalable SPD approach for CP-Ti.

SAMPLE PREPARATION

For the research, commercially pure Titanium was used. Table 1 shows the chemical composition of CP-Ti, which corresponds to ASTM B 348-95 Grade 2.

The initial surface hardness and yield strength of CP-Ti Grade 2 across the width direction were measured to be approximately 120–125 HV and 250–260 MPa, respectively. For the RCSR method, the specimen was cut into sheets with dimensions of 70 x 60 x 2 mm. The discontinuous RCSR facility was constructed for this investigation after several passes culminated in both corrugation and straightening. The corrugated roller had many teeth on top, and the bottom of the rig had a standard straightening roller. On top, there's a corrugated roller with many teeth, and on the bottom, there's a standard straightening roller. The experiment was carried out at room temperature, which prevents dynamic recovery and thus increases grain refinement. Each sample was placed between the roller gears, which were turned by a motor at a speed of 700 rpm. The specimens were initially bent when passing through a corrugated roller, and then straightened after passing through a traditional roller. The samples were processed for a variety of passes, including 1, 3, 6, 10, 12, 14, 17, 21, and annealing condition. (CP-Ti) Annealing, (CP-Ti)0P, (CP-Ti)1P, (CP-Ti)3P, (CP-Ti)6P, (CP-Ti)10P, (CP-Ti) 10P-Annealing, (CP-Ti)12P, and (CP-Ti)14P were the sample's designations as descripted in table 2. Following RCSR, the specimens were checked for microhardness along the longitudinal axis with a Vickers microhardness using a Vickers Indenter. For each 5 mm size, the hardness value was determined using 0.5 kgf and a dwell time of 10s for each measurement.

Table 1. Chemical Composition of commercially pure Titanium.

Element	**Fe**	**O**	**C**	**H**	**N**	**Ti**
Composition (weight %)	0.30	0.23	0.07	0.015	0.02	99.36

Table 2. Sample Designation with description.

Sample designation	**Description**
(CP – Ti)0P	Commercially Pure Titanium – Raw specimen
(CP – Ti)1P	Commercially Pure Titanium – RCSR after 1 Pass
(CP – Ti)3P	Commercially Pure Titanium – RCSR After 3 Passes
(CP – Ti)6P	Commercially Pure Titanium – RCSR After 6 Passes
(CP – Ti)10P	Commercially Pure Titanium – RCSR After 10 Passes
(CP – Ti)10P-Annealing	Commercially Pure Titanium – RCSR After 10 Passes – undergone Annealing process
(CP – Ti)12P	Commercially Pure Titanium – RCSR After 12 Passes
(CP – Ti)14P	Commercially Pure Titanium – RCSR After 14 Passes

RESULTS AND DISCUSSION

Microstructural Characterization

Figure 1 depicts the microstructure of deformation as a function of the number of passes. The number of passes and shear bands grows as the number of passes grows. Each shear band runs in the same direction as the shear band course. The shear band penetrates deeper into the grain as the number of passes increases. The shear band designated a 45° angle as Rolling Direction (RD). The number of shear band observed in more as the number of RCSR passes increased, going from 10% in the first pass to 40% after ten passes. For HCP materials like CP-Ti, the primary mode of deformation is slip along the basal plane. Deformation twinning, which is achieved by the formation of elongated grains and shear strain, is the primary deformation mechanism for material strengthening in CP-Ti due to the limited number of slip. Twin and slip were activated for grain refinement due to deformation, resulting in heterogeneous microstructure size as shown in figure 2 -3. This effect was also found in studies of the literature. [27]. The deformation twin density increases with rising strain after a certain number of passes and varies from one grain to the next. [18]. Furthermore, as the number of passes increases, the deformation increases. Owing to the small number of passes, a low angle grain boundary is initially visible. After 14 passes, the low angle boundary forms, resulting in subgrain formation. If the number of passes increases, the average misorientation angle increases, causing the grain refinement mechanism to turn low angle grain boundaries into high angle grain boundaries. [25]. As a consequence of various severe plastic deformation methods, many materials undergo these transformations.

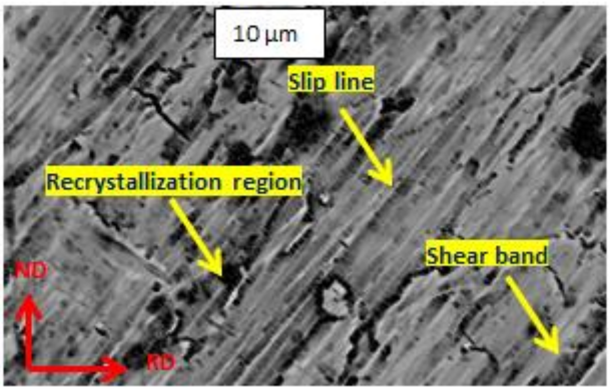

Figure 1. Single pass CP-Ti.

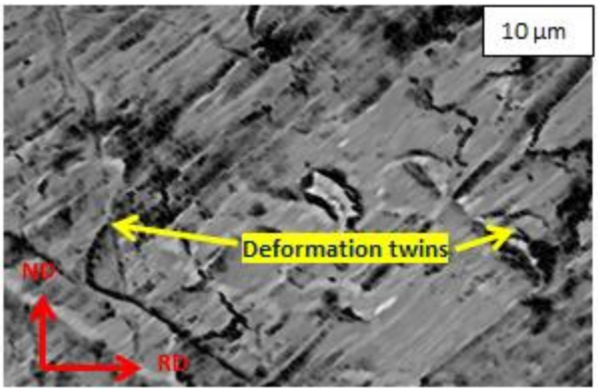

Figure 2. After 6 pass CP-Ti.

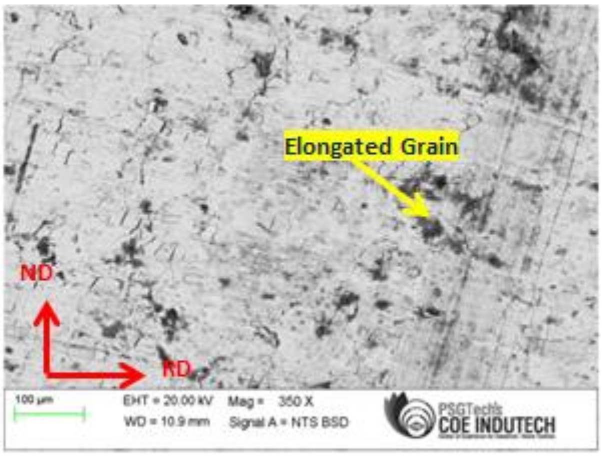

Figure 3. After 10 passes of CP-Ti.

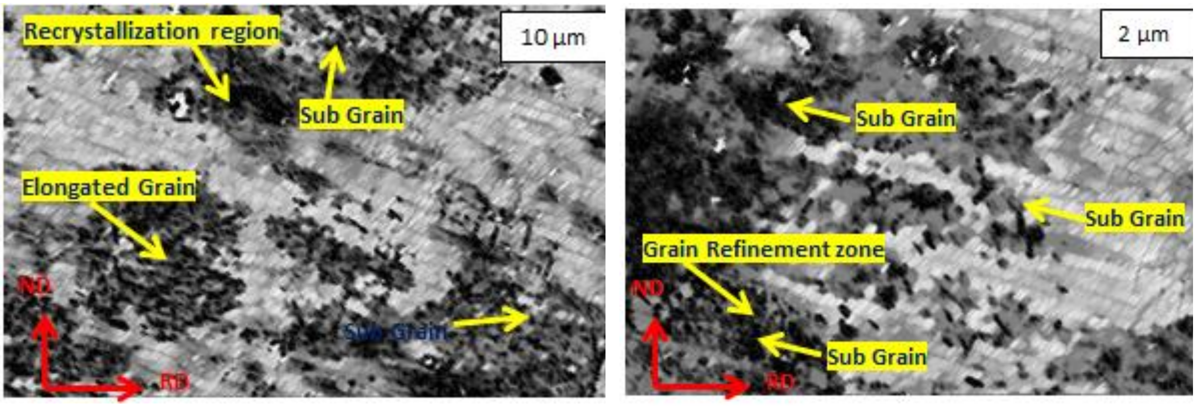

Figure 4. After 14 passes of CP-Ti.

Severe plastic deformation causes the recrystallization process, which leads to the grain refinement process. During the initial passes of this analysis, the dislocation densities increased, resulting in further plastic deformation and subgrain formation. As the passes were increased, more subgrain formation was observed, and the coarse grain structure was reduced to a fine grain structure due to the dynamic recrystallization process. Figure 4 shows how the elongated grain boundaries were broken into sub grain formation. The intermediate annealing process also altered the microstructure, making it easier to perform subsequent passes.

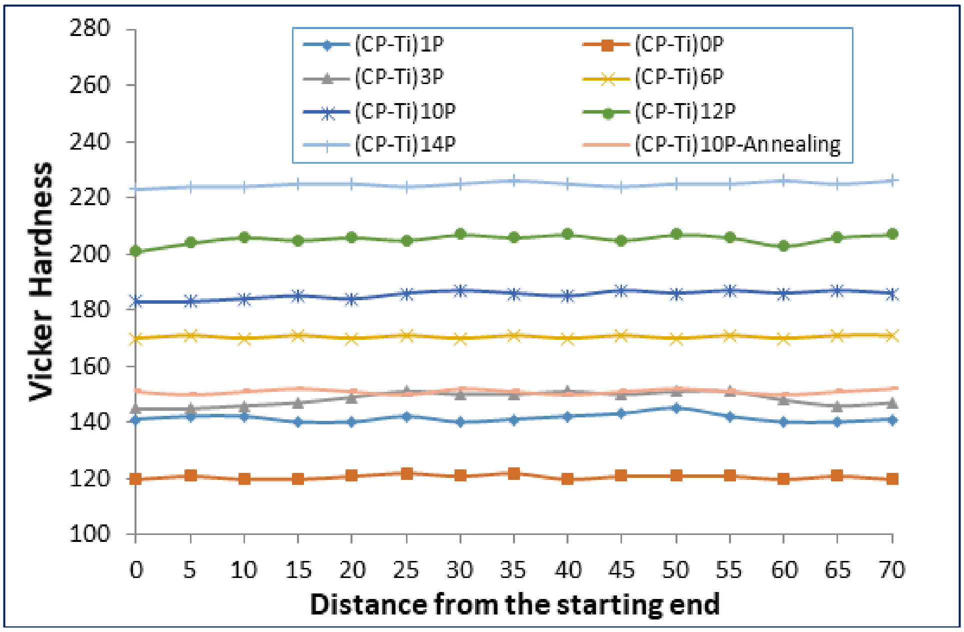

Figure 5. Variation of Hardness with respect number of passes.

Effect of Number of Passes on Hardness

The research looked at the toughness of the cast and the number of passes after RCS CP-Ti. Figure shows the Vickers Hardness, Hv, value for the RCS processed sample after a certain number of passes. Figure 3. against the longitudinal distance of 5 mm between two locations along each sample with the maximum distance of 70 mm. Due to the regularity of the corrugations placed on the sample, limited undulation of the hardness value is observed in the sample with higher passes. Owing to the initial extreme deformation on the specimen, there is a major undulation in the hardness of the sample, such as no passes and 3 passes specimens. After 10 passes in RCS, the specimen's hardness rises from 120±2 Hv in the cast condition to 185±2 Hv condition. As adapted in RCS, the percentage of micro hardness improvement after 10 passes is about 54% when compared to base materials. After 10 passes, the substance hardened even further, necessitating intermediate annealing to soften it. The temperature for annealing was set to 500°C. During the intermediate annealing process, the material underwent complete recrystallization, allowing for further material deformation. During the recrystallization process, the nucleation and grain growth rate are the key mechanisms for obtaining the recrystallized grain. Equiaxed grain is formed as a result of the recrystallization process. The micro hardness value of CP-Ti reached a limit of 224±3 after 14 passes of RCS processing. CP-Ti, on the other hand, had a cumulative microhardness of 203±2. 2 after two Groove pressing passes [28]. As compared to the base metal without RCS adaptation, the microhardness value increases by around 87 percent for 14 passes processed by RCS. The refining of grain, as seen in the microstructure, is responsible for the increase in hardness value. It is evident that grain refinement becomes more pronounced with an increasing number of passes, contributing significantly to the enhancement of hardness. This relationship can be quantitatively explained using the Hall–Petch equation, which correlates grain size (d) with yield strength (σ_y).

$$\sigma_y = \sigma_0 + k.d^{-1/2}$$

The grain refinement shown in the microstructures was connected with the microhardness's progressive rise with the number of passes. The hardness rises as the average grain size decreases as a result of deformation-induced twining, sub-grain formation, and ultimately recrystallization, according to the Hall–Petch equation mentioned above. The grain boundary strengthening mechanism is further validated by the transition from low-angle to high-angle boundaries, which becomes apparent after 14 passes. Numerous studies have documented hardness enhancement in CP-Ti through various SPD techniques. A comparison of the present results with those studies highlights the effectiveness and novelty of the RCSR approach employed in this work as listed in Table 3.

Table 3. Comparison with previous SPD studies.

SPD Method	No.of Passes	Final Hardness (Hv)	Reference
ECAP	8	~210	[32]
HPT	N/A	~240	[33]
CGP	2	~203	[28]
RCSR (our work)	14	224	-

Table 4. Hardness value before Annealing.

Condition	Average value of Hardness reached
CP-Ti - No Pass	120
CP-Ti - 1 Pass	140
CP-Ti - 3 Pass	145
CP-Ti - 6 Pass	170
CP-Ti - 10 Pass	185

Table 5. Hardness value after Annealing.

Condition	Average value of Hardness reached
CP-Ti - 10P – Annealed sample	152
CP-Ti - 12 Pass	200
CP-Ti -14 Pass	224

The refining of grain, as seen in the microstructure, is responsible for the increase in hardness value. Furthermore, CP-Ti reacted well to RCS processing, with substantial hardness improvements up to 14 passes. The hardness value before and after annealing process with multiple numbers of passes listed in Table 4 & 5. The attempt to proceed after 14 passes, i.e. 16 passes, was unsuccessful because it resulted in the development of surface cracks and further processing failure in the specimen.

CONCLUSION

The RCSR process was successfully used to produce commercially pure titanium for up to 14 passes. After 14 passes, the highest microhardness was reached. The maximum microhardness of 224±3 Hv was achieved after 14 passes. Furthermore, after 14 passes, the microhardness rose by 87% in comparison to the base metal. This notable improvement was ascribed to the gradual subgrain creation, deformation twining, and grain refining, all of which were more intense as the number of passes rose. An intermediate annealing step at 500ºC was added to promote dynamic recrystallization, which prevented fracture formation and allowed for up to 14 passes, allowing for greater deformation beyond 10 passes. This study's foundation is the methodical application of RCSR with a higher number of passes to CP-Ti, a comparatively untested SPD technology. For improving mechanical properties, RCSR provides a more straightforward, affordable, and scalable option than more intricate methods like ECAP and HPT. Overall, this work supports the Hall–Petch link by clearly demonstrating a relationship between microstructural evolution and hardness enhancement. According to the results, RCSR has great promise for usage in medicinal and aeronautical applications where fine-grained structures and increased surface hardness are essential.

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