Investigation of Mechanical Properties for Commercial Purity
Titanium Severely Plastic Deformed by Accumulative Roll-bonding Process

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Abstract
In this study accumulative roll bonding (ARB) process up to 8 Cycles (equivalent strain of 6.4) at ambient temperature was used in order to improve the mechanical properties of Commercial purity titanium (CP-Ti). Several experiment has been done and Mechanical properties of specimen has been discussed. For investigating optical microscope punching and hardness test were utilized. This is the first study on shear strength of CP-Ti formed by the ARB process. In this study Yield Shear Strength (YSS), Ultimate Shear Strength (USS), elongation, punching energy and hardness were investigated. Results of experiment report that as the number of the ARB process increased, the shear strength increased but after that certain cycle YSS and USS gradually decreased. As well as increasing numbers of ARB cycles, elongation decreased. Result reported that the shear strength of specimen reached to maximum value at certain cycle and afterward due to grain recovery and specimen fracture, it decreased. Experiments indicate that maximum hardness’s and shear strength were obtained in cycle 4 and 6 respectively. Furthermore specimen fracture after 8 cycles ARB processing was observed.

Keywords
Accumulative Roll Bonding, Titanium, Punching Test, Shear Strength

1. Introduction
Commercial purity titanium is one of the most important metals in aerospace, biological applications and industrial purposes. It has H.C.P crystal structure, the most useful attributes of the titanium and its alloys are corrosion resistance, excellent toughness, biocompatibility and the highest strength to density ratio. It has been already established that severe plastic deformation (SPD) can realize the ultra fine grained (UFG) in metallic materials [1]. The formation mechanism of the UFG has been an issue of contention, and many researchers have studied the microstructure evolution during SPD. One of the ideas to explain the microstructure evolution during the SPD process is grain subdivision [2, 3]. The grain subdivision in SPD is a process where deformation induced boundaries subdivides the original crystals as well as the deformation induced boundaries, have been classified into: geometrically necessary Boundaries (GNBs) and incidental dislocation boundaries (IDBs) [4]. In the ultrahigh strained materials; a number of GNBs are introduced to form the UFG microstructures. The grain subdivision has been systematically studied in F.C.C. metals
and alloys in which slip systems can be strictly determined. On the other hand, the SPD processes have been applied primarily for metals and alloys having cubic crystal structure, and the number of studies on SPD of hexagonal materials is limited. It is well known that deformation behaviors of cubic metals are significantly different from those of hexagonal metals. Unlike the cubic crystal structure, in hexagonal metals number of active slip systems is limited, and sometimes deformation twinning plays an important role for plastic deformation. Therefore, it is anticipated that the microstructure evolution during SPD is different between cubic and hexagonal metals.

From an industrial point of sight, it is furthermore desirable to raise great quantities of bulk material. In this context, ARB process seems to be very promising for continuous sheet production on established large-scaled manufacturing facilities [5-7]. Moreover, it was testified before that ARB leads to a significant increase in the mechanical properties, especially concerning strength and strain rate sensitivity [8-9].

ARB has previously been applied to grain refinement of commercial purity Ti; however, there is very trivial detail in the literature on ARB Benefits of ARB includes the role of conventional roll forming equipment and the ability to produce bulk UFG sheets with dimensions limited only by the capability of the coils. ARB involves stacking two or more plates of metal, and rolling them together, in order to refine the grain structure and cause them to adhere through the roll-bonding process. The rolled sheet is then sectioned in half, degreased, stacked and the procedure repeated (Figure 1). One of the features of accumulative roll-bonding is that it is possible to impart large plastic strains on the materials over a number of ARB cycles. A modified ARB rolling process was developed, involving an initial cold roll at ambient temperature in order to sever deformation and break up the coarse grain structure.

However, the ARB mainly has been used for cubic materials and the number of researches used to try applying ARB to H.C.P. metals is limited. D. Kent et al. tried to form titanium by appalling the ARB process. They found that After 4 cycles (8 layers), the ARB-processed sheet exhibit eda UTS of 1220 MPa, 0.5% proof stress of 945 MPa, ductility of 4.5% and uniform elongation to 3.0% strain. Furthermore, they observed UTS around 70% higher and the proof stress almost double that of the coarse-grained solution treated alloy [10].

In the other investigate of CP-Ti processed by ARB at ambient temperature, by Terada et al. [11], they reported an equiaxed microstructure with improve of tensile strength of 890 MPa after six ARB cycles at ambient temperature. A similar strength improvement in tensile test was obtained in CP-Ti processed at room temperature using high-ratio differential speed rolling (HRDSR) conducted by kim and Yoo [12]. Room temperature equal channel angular pressing (ECAP) of another process to SPD of CP-Ti has been carried out by increasing the die angle from 900 to 120–1351, thus decreasing the strain reported in per pass. Xzhao et al. [13] demonstrated that enhancing the die angle to 1201 improved the tensile strength up to 780 MPa in just one pass. Furthermore, Yzhang et al. [14] processed CP-Ti by ECAP with a die angle of 1351 and proved that the tensile strength was increased to 750 MPa in two passes. It is interesting to note that these values are only slightly higher than the strength observed in CP-Ti that had been processed by SPD in 7–8 passes at elevated.

The purpose of the present study is to clarify the change in mechanical properties (shear strength and hardness) in pure titanium (CP-Ti) during SPD. In this study, a commercial purity Ti (CP-Ti)
severe plastic was deformed by applying the accumulative roll bonding (ARB) process. These studies showed the Micrograph of ARB specimen after SPD, but the microstructure evolution during SPD are still unclear due to lack of systematic observations. The ARB is an SPD process using rolling deformation, and it is applicable to continuous production of sheet materials [10, 21]. The ARB has been applied to various kinds of metals and UFG microstructures were successfully obtained. However, the ARB mainly has been used for cubic materials and in this trial it was tried to apply the ARB to H.C.P. metal. The predesigned SPD technique for this investigation was ARB. Here we demonstrate the successful use of the modified ARB process to form commercial purity titanium with enhanced shear strength and hardness. Details of the evolution of the microstructure and mechanical properties of Ti-CP specimens over several ARB cycles are presented. Results from optical microscopy, hardness test were obtained in conjunction with mechanical property data from punching test. Comparisons are drawn to microstructures and properties obtained for 8 cycles of ARB process.

2. Experimental Tests
A CP-Ti (ASTM grade 2) sheets 0.5 mm in thickness, 50 mm in width and 200 mm in length were used in this study. The chemical composition of the specimen (CP-Ti) is shown in Table 1. The starting sheets were firstly deformed to 50% reduction in thickness (equivalent strain of 0.8) by ARB process. This procedure is hereafter considered as the first ARB cycle. The 50% cold-rolled sheets 0.5 mm thick were cut in half-length, stacked to be 1 mm thickness after degreasing surface of the sheets by using chemically cleaned acid solution between each cycle and wire-brushing the contact surfaces, and then roll-bonded by 75% total reduction in one pass, which is considered as the second ARB cycle. The procedures in the second ARB cycle were repeated up to 8 ARB cycles (total equivalent strain of 6.4). The ARB was carried out under well-lubricated condition using a two-high mill having a roll diameter of 220 mm. The Roll peripheral speed was 10 m/min. The specimens were cooled in water after each ARB cycle. The micrograph characterizations of the CP-Ti sheets ARB-processed by various cycles (various strains) were carried out by optical microscope (OM). The observed sections for image were perpendicular to the normal Direction–Rolling Direction (RD-ND) of the sheets. The observed plane was electro-polished in 50 ml HClOO solution. Orientation analysis of shear punching tests conducted for the ARB-processed specimens were parallel to ND.

Small disc specimens of 20mm diameter and 0.5 mm thickness were electric discharge machining (EDM) wire cut from the various surfaces were gently ground using SiC 600 grit to a final thickness in the range of 0.5±0.005 mm. Shear Punch tests were executed utilizing a universal test machine at room temperature and at a constant crosshead speed of 0.2mm/min. Tests were conducted on specimens to study the effect of different number of ARB process on the punching test result. Three samples were punched for each test and the average values are reported.
3. Results

Figure 2 shows microstructures of the CP-Ti specimen before the ARB starting material equiaxed grains with mean grain size of 50µm. Figure 3 illustrates the optical micrographs of CP-Ti specimen 1 to 8 ARB process cycle in ND-RD direction. It is evident in the first cycle that the interfaces between two plates were easily recognizable (Figure 3a). It is likely to be indicated that weak joint between two plate occurred however in the next cycles 2-4 with increasing ARB cycles, interfaces of the previous layer disappeared (Figure 3b, 3c and 3d). This reflects that by increasing ARB cycles, the joint between the subsequent bonding of interfaces improved, although still weak joint in fresh layer can be determined. After 4 cycles, interfaces of layers were observed more integrated (cycle 5-6) (Figure 3e and 3f). The number of layers achieved by 2 should be noticed where n is
number of ARB process cycles. Figure 3 shows the CP-Ti specimen in the last cycles of experiment (cycle 7-8) its evidence increment in ARB cycles, resulted specimen fracture.

Figure 2. Microstructures on longitudinal sections of the commercial purity Ti

Figure 3. Micrograph images of the ARB sheet viewed from TD and ND. (a) cycle 1, (b) cycle 2, (c) cycle 3, (d) cycle 4, (e) cycle 4, (f) cycle 5, (g) cycle 6, (h) cycle 7, (i) cycle 8
3.1. Shear Punching Test
In order to characterize mechanical properties of the specimens, the shear punch test (SPT) was used. The (SPT) technique involves slow blanking of a thin disc of specimen clamped between a set of die and punch at a constant speed as shown schematically in Figure 4. The deformation occurs in the small annular region of the punch–die clearance. The load on the punch is calculated as a function of specimen displacement. The curves obtained from the SPT data are similar to those in a tensile test. This test has been designed for determination of shear properties of the specimens, therefore, yield shear stress (YSS), and ultimate shear strength (USS) can be measured, using the following experiential equation[15].

\[ \tau = \frac{p}{2\pi rt} = c\sigma \]  

Where \( p \) is the applied load, \( r = (r_{\text{punch}} + r_{\text{die}}) \), \( t \) is specimen thickness, \( C \) is a correlation coefficient, \( \tau \) and \( \sigma \) are the estimated corresponding shear and tensile yield or maximum stress, respectively. Leon and Drew et al. [16] And also Rguduru et al. [17] established a linear relationship between the shear and tensile data for yield and ultimate strengths, briefly put, they are:

\[ \sigma_{0.02} = \alpha \tau_{0.02} \text{, Where } \alpha = 1.77 \]  

\[ \sigma_{\text{UTS}} = \beta \tau_{\text{USS}} \text{, Where } \beta = 1.8 \]  

Moreover, Toloczko et al. [18] proposed other correlation between the strain hardening exponent obtained from the shear punch testing data and the uniform elongation, briefly put, they are:
0.002

\[ \frac{\eta}{0.002} = \frac{\tau_{\text{US}}}{\tau_{\text{SS}}} \]  

(3)

And

\[ e = 2.26\eta - 0.15 \]  

(4)

Where \( e \) is the uniform elongation, \( \eta \) is the strain hardening exponent in SPT test, \( \sigma_{0.02} \) and \( \tau_{1.00} \) represent the estimated 0.02% tensile and the 1.00% SPT normalized displacement offset yield point, respectively.

The punching energy of shear punching is calculated by [19]:

\[ E_p = \int_0^{\delta} \tau d\delta \]  

(5)

Where \( E_p, \delta \) are \( \tau \) punching energy, displacement and shear stress respectively. In the other word, the total punching energy is taken as the area under a given shear stress – displacement curve (SDC). It is obtained during the blanking operation and is very similar to that acquired by a conventional uniaxial tensile test. The properties obtained by analyzing the SDC can be correlated to the corresponding conventional tensile properties. After application of the load, the applied load \( P \) was measured automatically as a function of punch translocation; the data were obtained by a computer so as to define the shear stress of the tested materials using the Equation 1. Figure 5 shows SDC to specimens shaped by ARB process and the stress variations calculated during the punch test until blank separation. For better comparison shear stress – displacement curve for cycles 2, 4, 6 and 8 is showed in Figure 5.

![Figure 5. Comparison of shear stress –displacement curve of different ARB cycles](image)

It shows nominal shear stress – displacement curve of the CP-Ti sheets ARB-processed by various cycles. The shapes of the SDC were completely different between the first cycle and the last cycle of ARB-processed specimens. The strength, elongation and punching energy obtained from the curves are summarized in Figure 6 to 9, a function of the number of ARB cycle. Figure 6 illustrated
Yield Shear Strength (YSS) of the CP-Ti ARB-processed by various cycles at ambient temperature. The high YSS is observed in cycle 4 (400 Map).

Figure 6 illustrates the Ultimate Shear Strength (USS) of the commercial purity Ti ARB-processed for 8 cycles. As specified in the graph, ARB process improves the strength of a specimen up to 560 Mpa in cycle 4 and a substantial reduction can be observed after that. Similar behavior of titanium alloy specimen in tensile tests of ARB process has been reported by Terda et al. [11] and Kent et al. [10] With the exception that the high strength respectively in cycle 6 and 3 archived. The lowest strength which was observed in cycle 8 was about 420 Mpa and by comparison Figure 3 with Figure 7 and 8, it can be deduced that the ARB specimens fracture of final cycles play impressive role in specimen strength.

Figure 7. Ultimate Shear Strength (USS) of the commercial purity Ti ARB-processed by various cycles
Figure 8 depicts total shear elongation of specimen in various ARB cycles. According to the diagram in the first and second cycle, the percentage of elongation is about %13-%15 and also the maximum elongation is observed in those cycles, but after second cycle the elongation also significantly dropped and gradually decreased by increasing the ARB cycle, the elongation decreased from the second cycle to last cycle about 0.2%. By comparing the measure of shear elongation and tensile elongation, it can recognize that there is a tight relationship between CP-Ti shear elongation and tensile elongation [11].

The energy to separate blank of specimen in punching test is calculated by using Equation 5 and is shown as a function of punching energy in Figure 5. The data show a significant increase around the cycles 2 by value of 0.11 J. It is because the large elongation occurred in the cycle of Figure 8 and according to Equation 5 punching energy is increased. On the other hand, it does not show a significant change of the punching energy, in 6 subsequent cycles.
3.2. **Hardness Test**

The hardness of the rolled specimen was measured, and the averages of the measurements are shown in Figure 10. The average Vickers hardness, obtained using a force of 5kg, is given on the ordinate and the number of layers rolled is shown on the abscissa. As expected, the hardness increases as the number of passes is increased. The hardness of the first cycle is 160 HV. At the end of the six pass, after rolling the 64-layered strip, the hardness has become 318 HV and then decreased gradually. It is observed that the largest increase in the hardness, nearly 70%, is created when the four-layered strip was rolled (cycle 2). The hardness of specimen enhanced in the subsequent passes, but at a progressively lower rate.

![Figure 10. Changes in Vickers hardness of the commercial purity Ti ARB-processed by various cycles](image)

### 4. Discussion

The change in micrograph and mechanical properties of a CP-Ti during SPD by ARB was clarified in details in the present study. It was found that by applying ARB process, shear strength enhanced and at specific cycle it reaches to a maximum value of strength and then gradually reduced. It has been reported by many investigators that the sharp rise in strength at relatively low strains is mainly due to the work hardening caused by an increase in dislocation density and formation of the subgrains. The low density of dislocations at relatively high strains is because of the dynamic recovery [20] or absorption of dislocations into the grain boundaries [21]. Many researchers investigated microstructure during SPD and reported equiaxed grain by increasing SPD [11]. It is well known that deformation twining plays an important role for plastic deformation in H.C.P. metals that have limited number of active slip systems. Also tensile twin and compressive twin were reported in the deformed samples [22]. The twin boundaries formed at lower reduction changed to normal high-angle boundaries owing to lattice rotation during plastic deformation [11]. Tereda et al. [11] indicated that twinning did not occur during deformation process at high strain. Hence, the deformation twinning has little influence on the ultrafine microstructure evolution in the specimens ARB-processed by many cycles.

It should be mentioned that the thermal conductivity of Ti is especially low (17 W m⁻¹C⁻¹) compared with other metals, that is, significant increase in local temperature is expected within the
shear bands in CP-Ti, which assists the recovery of the regions having high density of defects to form equiaxed grain structures. It seems, therefore, reasonable that the equiaxed grains are formed within the shear bands because of enhanced recovery during the process.

In any way, the increase of the volume fractures Figure 3 of specimen by increasing in ARB cycle and local adiabatic heating that assists recovery during SPD to form the equiaxed grains [11]. It is due to reduction of strength and hardness in last ARB cycles.

Significant recovery of ductility in conjunction with continued improvement in strength is not normally observed for accumulative roll-bonding processed materials, which normally display monotonous reductions in ductility in association with strength enhancements for increasing numbers of cycles [2]. Wkim et al. [12] did report decreasing elongation with increasing numbers of ARB cycles in an 8011 Al alloy, accompanied by reduced strengths and hardness. It was suggested that this was associated with enhanced dynamic recovery during testing. Other authors [23,24] also reported a significantly drop in the elongation followed by some recovery with subsequent cycles in accumulative roll-bonding processed copper and 70/30 brass strip, respectively. Although, neither clarified whether this was due to enhanced elongation, nor gave an explanation for the recovery. Similar to the CP-Ti, the strength of the 70/30 brass strip and copper material also continued to improve in conjunction with the recovery of elongation.

5. Conclusion
The accumulative roll-bonding process up to 8 cycles has been performed successfully for a ti-cp. The results obtained are summarized as follows:

1- By increasing layer in ARB process, the interface of previously layer is improved.
2- The shear strength of the ARB processed CP-Ti increases with the number of ARB cycles and in the certain cycle reach to maximum value, so that in this study the maximum value of USS and YSS achieved after 4 cycles, by amount of USS and YSS respectively 560,405 MPa.
3- The elongation decreases abruptly after one cycle, and it decreases slightly with the number of ARB cycles and it holds straight relationship with fracture energy.
4- Hardness increases were observed after each accumulative roll-bonding process cycle, but results showed some recovery with increasing cycles. The largest jump in hardness was achieved after the first processing cycle, which suggests that the most effective ARB processing is gained at low cycles.
5- With excessive ARB cycles (more than 7 cycles) specimen fracture occurred and it plays an impressive role in the mechanical properties of specimen.

6. References


