

Effect of TiO₂ nanoparticles on the bond strength of CP Ti sheet by cold rolling method

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ABSTRACT

In this work, the strength of the cold roll bond of the commercially pure titanium (CP Ti) sheet and the effect of TiO₂ nanoparticles on the bond strength were investigated. Through the accumulative roll bonding process, which is considered one of the important severe plastic deformation processes, the creation of suitable bonds can play a significance role in the ultimate strength of the bond. Thus, at first the optimum bonding parameters of two or several metals producible via ARB method were studied and then manufactured. In the present work, the bond strength of the commercially pure titanium sheets was investigated. Based on the results of the peeling test for CP Ti it was found that by applying a 50% thickness reduction, a desirable bond strength was achieved. To study the effect TiO₂ nanoparticles on nanocomposite preparation in the ARB test, these nanoparticles were dispersed between the CP Ti sheets at 0.1, 0.3 and 0.5wt% concentrations. The results showed a decrease in the bond strength during the peeling test by the increase in the TiO₂ content. The scanning electron microscopy (SEM) tests of the peeled sites revealed that the presence of cumulative layers of nanoparticles prevented bonding in these areas. Although the addition of TiO₂ nanoparticles resulted in the reduced bond strength, at TiO₂ percentages equal or less than 0.5wt.%, this decrease was negligible. Hence, the ARB method could be considered as an efficient technique for the preparation of the bulk composite at room temperature.

1-Introduction

Solid-phase welding can be used for manufacturing automotive parts, bimetal products and household items. Therefore, understanding the mechanisms and the details of the process can be significantly important in industries. This welding technique can be used on a large variety of materials with the same attributes or with different mechanical and metallurgical properties. Roll welding is a solid-

phase welding process through which bonding is achieved between the contaminant-free areas as well as the virgin metal surfaces that are exposed and extruded through the cracks of the fractured covering layer due to plastic deformation [1-3]. Titanium and its alloys are well-known materials with widespread applications. Over the recent decade, the focus of studies on these strategic alloys has been on their mechanical properties [2-5, 7-10], corrosion [11] and creep

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[12–14] behaviors. Recently, severe plastic deformation (SPD) processes have been applied as powerful techniques for reaching high strength metals by mechanical working and grain refinements. These methods allow manufacturing bulky metals to nanostructure bulky specimens. The accumulative roll-bonding (ARB) process is a well-known SPD method applicable for manufacturing bulk industrial specimens. The bond strength at the initial ARB cycle is an important production parameter for materials manufacturing. In this regard, investigation of good cold bond strength is important for ARB processing.

For very hard particles such as SiC [15–18], B₄C [19] and others severe cracking of the particles during deformation process of composites has been reported. The cracked particles cannot effectively tolerate any load and act as voids. Thus, there is reduction in the strength of the composites compared to monolithic materials. Karimi and Troghinejad [18] showed that despite the mean SiC particle size of 10.5 μm, severe cracking and breaking of the particles were observed mainly in the clustering areas. In these areas, the ceramic particles were in contact with each other, exerting high stress into particles during deformation which, in turn, caused cracking and breakage. Also, the shape of ceramic reinforcement was important as the sharp corners of particles resulted in cracking in the matrix. However, in the case of using ceramic particles similar by matrix such as Al₂O₃ in Al matrix [20–22] or TiO₂ in Ti matrix [23] and so on cracking and other problems have been reported.

Improvement of reinforcement distribution with increasing the number of ARB cycles has also been reported in other studies [20, 23–24]. Jamaati and Troghinejad [26] investigated the microstructure of Cu/15 vol.% Al₂O₃ composite produced by the ARB process. They showed large particle free zones and big agglomerated and clustered particles in the fifth cycle and a homogeneous distribution and good reinforcement/matrix bonding without considerable clusters. Also, they demonstrated that porosities and the particle free zones were not observed at initial ARB cycles when

microparticles employed [25]. Therefore, we utilized TiO₂ nanoparticles in the present study. Mechanical properties of titanium alloys can be improved by reinforcing them with ceramic particles [18–22]. In previous works, different particles such as TiC [27], TiN [28], and SiC [18, 22–27] were used as reinforcements for titanium alloys. TiO₂ establishes a high strength and stronger specific Young modulus and, as a consequence, it is nowadays widely used as reinforcement [28, 29]. In this investigation, the ARB process was used as an effective alternative method for manufacturing high-strength CP Ti/nanoTiO₂ nanocomposites. There has been no previous work on producing Ti matrix composite by ARB process. The aim of this study was to evaluate the microstructural and mechanical properties of monolithic CP Ti and CP Ti/nanoTiO₂ nanocomposites produced by the ARB process. TiO₂ nanoparticles were dispersed in titanium matrix during the process to use a combination of severe plastic deformation and second phase particles effects in material strengthening.

In the present study, for the first time, the bond strength between two kind of CP Ti is reported and the addition of the TiO₂ nanoparticles as the reinforcement factor in the cold roll is discussed.

2- Materials and methods

2-1- Materials

A commercial pure titanium (CP Ti) sheet with the thickness of 0.9 mm was used in the present study (Table 1). This sheet was cut into 15cm×4cm pieces parallel to the sheet rolling direction, and doped with TiO₂ nanoparticles as reinforcement.

2-2- Methods

2-2-1- The cold roll bonding (CRB) process

The schematic illustration of the ARB process for nanocomposite synthesis is shown in Fig. 1. In this study, titanium strips were ARBed within two steps. Firstly, for producing a satisfactory bond between two layers of titanium strips in cold roll bonding, it was essential to remove contaminations such as oil, grease, etc. on the joining surfaces of titanium sheets.

Table 1: Chemical composition of the CP Ti (grade 2) as-received sheet.

Elements	Ti	Al	Mo	Sn	Zr	Mn	V
Percent (wt%)	96.80	<0.015	0.649	<0.50	<0.100	<0.100	<0.100
Elements	Fe	Nb	Cr	Cu	Ni	Pd	
Percent (wt%)	<0.100	<0.50	<0.020	<1.0	<0.0030	0.195	

Acetone bath was used as a strong degreaser for the initial preparation of titanium sheets surface. In the second step, the scratches were brushed with a stainless steel rotating wire cup brush with 0.4 mm wire diameter. To avoid any oxide formation in two new surfaces influenced by scratch brushing, the roll bonding process had to be carried out immediately (part (1) in Fig. 1). After surface preparation of titanium sheets, TiO₂ nanoparticles were uniformly dispersed on the prepared surface of one sheet (part (2) in Fig. 1)

and then the other sheet was stacked over and fastened by copper wires at both sheets ends (part (3) in Fig. 1). The stacks were handled fast and carefully and the specimens were rolled as soon as surface preparation was completed (part (4) in Fig. 1). The roll bonding process was carried out with a 50% reduction (first step). The rolling machine used in this study had a 220 mm roll diameter working at a 4.5 m.min⁻¹ roll peripheral speed.

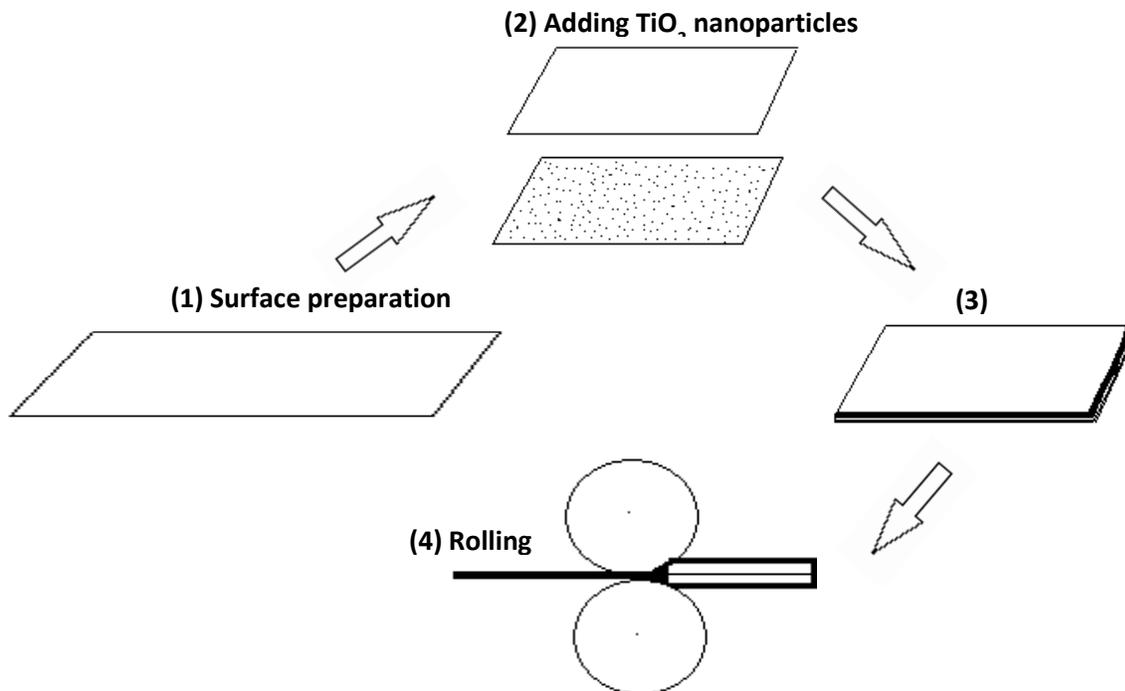


Fig. 1: The schematic illustration of the cold roll process for manufacturing the CP Ti/TiO₂ nanocomposite.

2-2-2- The peel test

By using a peeling test (Fig. 2), the strips were assessed for their bond strength according to ASTM-D1876-01. The peel tests were performed using a Hounsfield H50KS tensile testing machine with a 50 kg load cell and a crosshead

speed of 20 mm/min. In this test, the breaking-off forces were calculated from Eq. (1).

$$\text{Average peel strength} = \frac{\text{average load}}{\text{bond width}} \left(\frac{N}{mm} \right) \tag{1}$$

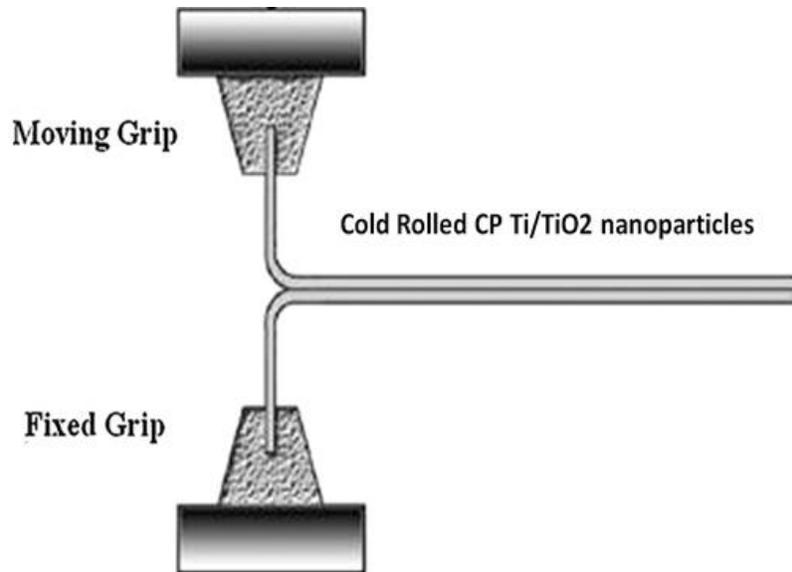


Fig. 2: The clamping configuration used in this study.

2-2-3 Microstructural evaluations

An optical microscope was used for studying the created as-received titanium microstructure. The samples were prepared by polishing with 600, 800, 1200, 4000 mesh polisher sheets. Also, Kroll's reagent (2ml HF, 4ml HNO₃, and 100ml H₂O₂) was used for etching the created microstructure. After peeling, the fracture surfaces of the samples were examined using a PHILIPS XL30 scanning electron microscopy (SEM).

3-Results and discussion

3-1- The as-received materials

TEM image of TiO₂ nanoparticles showed that the TiO₂ nanoparticles (Fig. 3) had a good distribution in size and good similarity in shape, with an average size of 15 nm. Fig. 4 presents the optical image of the microstructure of the as-received commercial pure titanium. This figure

illustrates the equiaxed and single-phase α -Ti with an average grain size of about 39 μ m. Also, a large number of twins were observed in grains, implying the crystal structure of Cp Ti with a hexagonal structure.

3-2-The bond strength of sheets in the peeling test and the effect of nanoparticles

Fig. 5 shows a force-distance diagram of the peeling test for the monolithic CP Ti and CP Ti with various percentages of TiO₂ nanoparticles and the same thickness reduction of 50%. This figure shows that the addition of TiO₂ nanoparticles led to a reduced peeling force in the titanium sheet, as adding TiO₂ nanoparticles up to 0.5wt.% resulted in a peeling force one-eighth of the nanoparticle-free state. Another noteworthy point in Fig. 5 is the fluctuations observed in the peeling force with increasing the nanoparticles content, as for the sample with 0.5wt.% TiO₂ nanoparticles, a severe variation of peeling force at low forces was seen.

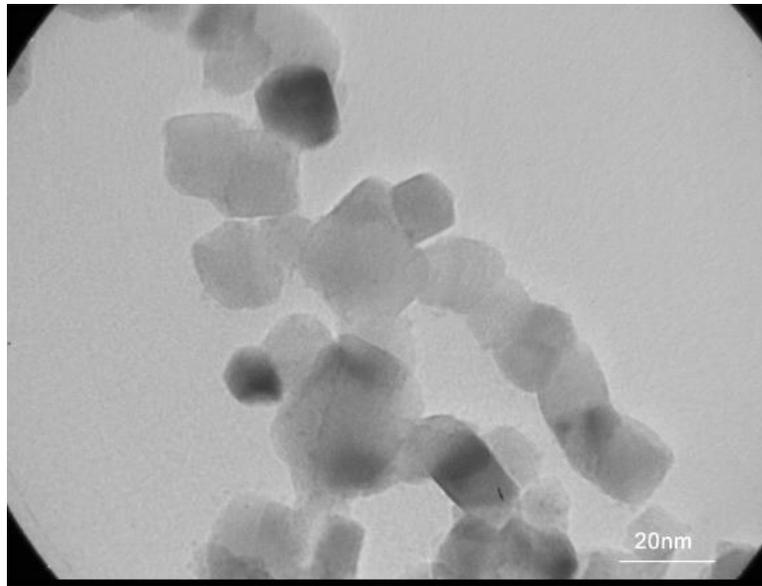


Fig. 3: TEM image of TiO₂ nanoparticles.

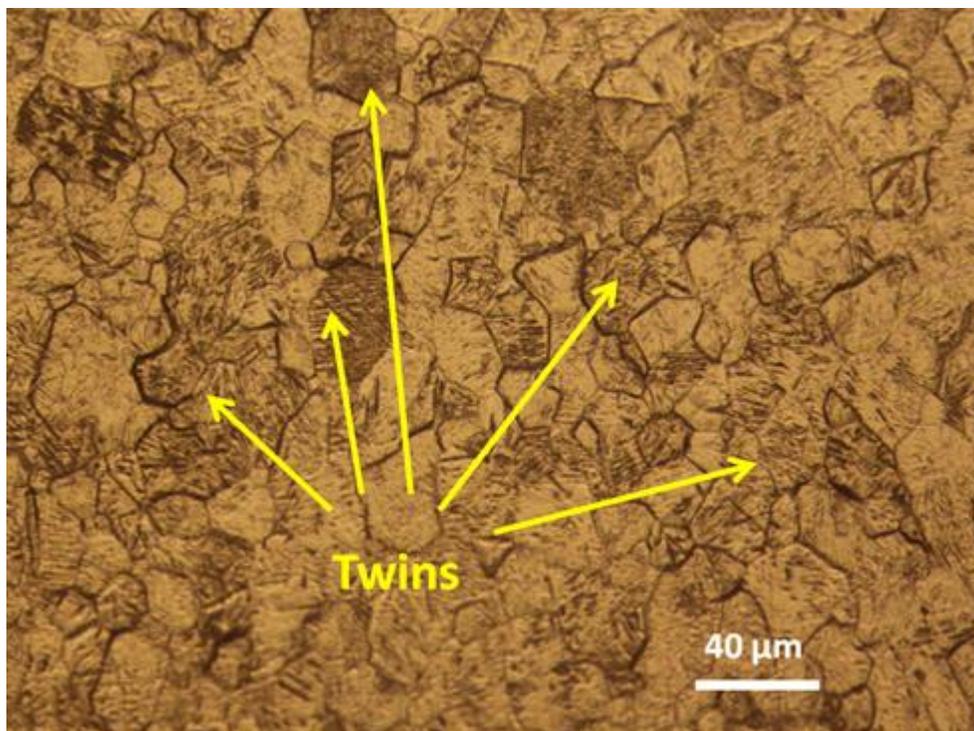


Fig. 4: The optical image of the microstructure of the as-received commercial pure titanium.

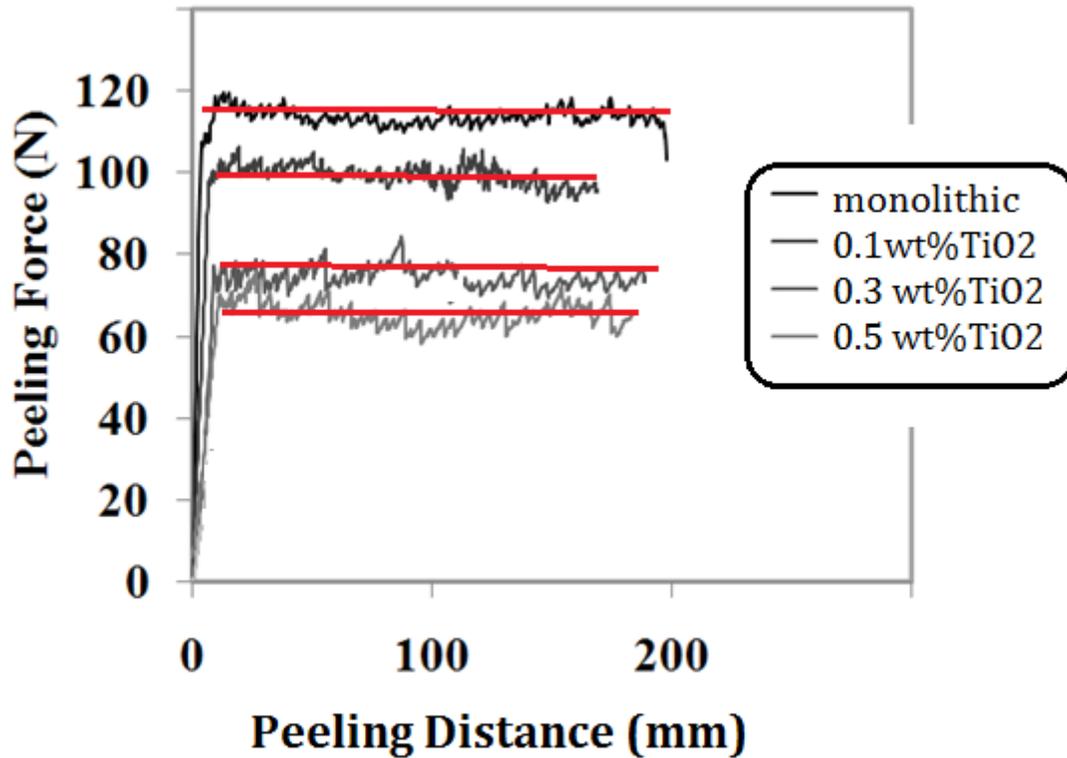


Fig. 5: The peeling test results.

The main explanation for the variations of the peeling force with increasing the TiO_2 nanoparticles content is the accumulation of nanoparticles in particular sites and lack of suitable bonds in these areas; this not only decreases the peeling force, but also leads to the increased variations of the peeling force. Figure 6 illustrates the SEM images of the peeled surface after the peeling test for different specimens. As it is demonstrated in this figure, the increase in TiO_2 nanoparticles content was accompanied by the expansion of the non-bonded areas.

The studies on bond development with the addition of reinforcing phases through the ARB process are mainly limited to aluminum. Such

studies have dealt with the addition of Al_2O_3 to Al 1100 and the assessment of the effect of different thickness reductions on the developed bonds [32]. In another study [33], the effect of micrometric TiH_2 particles on different thickness reductions was investigated, showing that the increase in the concentration of these particles led to a declined number of the developed bonds. In both of the abovementioned studies, the presence of nanoparticles prevented the direct contact of the pure metal extruded from the surface cracks in both metal surfaces in accordance with the oxide layer theory, finally resulting in the degraded bond strength of the two sheets. Other researchers have also reported similar results [30].

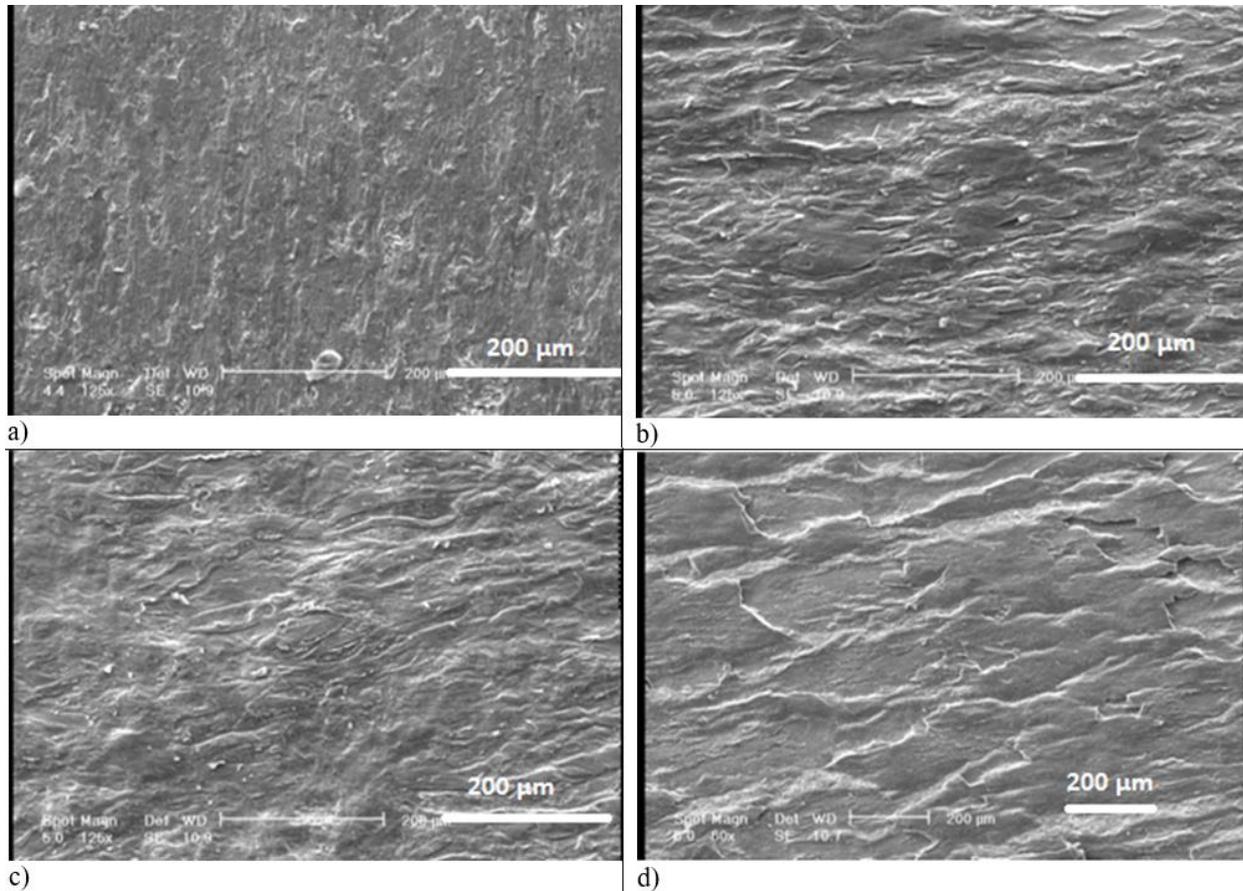


Fig. 6: SEM images of the peeled surfaces of samples including: a) the monolithic CP Ti; b) CP Ti/0.1wt.% nano-TiO₂; c) CP Ti/0.3wt.% nano-TiO₂; and d) CP Ti/0.5wt.% nano-TiO₂.

In the present work, the increase in the TiO₂ nanoparticles concentration was accompanied by the decrease in sites with bond and the creation of areas with weaker bonds or no bond. According to the oxide layer theory, which is the most recognized mechanism proposed for the ARB process [17-19], the oxide surface layers developed during the sample preparation and brushing are broken during the rolling process and the pure metals beneath the oxide layer, which is extruded under the exerted rolling pressure, are connected together and a cold welding is developed between the two layers. In the present work, it was observed that an increase in the TiO₂ nanoparticles content was required for peeling various ARB layers. In this regard, addition of the reinforcing nanoparticles phase through the ARB process could affect the mentioned mechanism in different ways:

a) When nanoparticles are placed between these two metallic sheets, a smaller number of cracks in

these two sheet surfaces are in direct contact with each other during the ARB process and a limited amount of metal from these two layers is connected together. Furthermore, it is known that nanoparticles generally have a much larger surface to volume ratio, as compared to microparticles; thus, this phenomenon is expected to be more severe in the presence of nanoparticles as the reinforcing phase.

b) By the introduction of nanoparticles between these two sheets, the pressure required for bond development between these two layers could increase. By adding these particles between the layers, the metal driven out from these two layers must be moved in larger distances for bond development. It can be concluded that the more this distance is within the equal thickness reduction, the weaker are the bond and the peeling force. Besides, an increase in the nanoparticles content could lead to the increased

distance between the extruded metals and a decreased bond and peeling force. This phenomenon could be more obvious by the addition of nanoparticles, rather than microparticles, because of the increased surface to volume ratio in the nanoparticles.

c) Shear deformation in the presence of particles finally resulted in the creation of further dislocations and metal hardening as well as the limited metal flow, ultimately leading to the decreased effective bond between two layers.

d) Lack of proper particle distribution between the layers could lead to peeling variation during the peeling test. As nanoparticles content between the layers increased, a less unified particle distribution was observed, leading to the nonunified bonding of these two layers during the ARB process and variation in the peeling force. It is worth mentioning that this phenomenon is more evident for nanoparticles rather than microparticles.

3-3- Bonding between the layers

In the present work, after the incorporation of nanoparticles with different percentages and bond development between titanium sheets, the prepared specimens were subjected to microscopic studies using RD-ND sheets. As shown in Figure 7, the optical microscopy images of the bonds between the rolled layers within the first passes of the process indicated an increase in the bonding distance of the two sheets as TiO₂ nanoparticle content increased. Figure 7a illustrates the monolithic (nanoparticle-free) CP TiO₂ specimen, which had a shorter bond distance between two sheet surfaces or, to put it in another way, the largest contact surface, as compared to those shown in Figs. 7b, 7c, and 7d. These figures

show a decrease in the contact area between the two sheets with an increase in the nanoparticles concentration. This observation confirms the results of the peeling test, indicating a reduced peeling force with increasing the nanoparticles content [17-21] (Fig. 5).

However, the studies conducted on SiO₂ nanoparticles added to Al6060 have indicated different results [37]. Researchers have shown that the presence of SiO₂ results in the enhanced strength of the bonds developed between aluminum layers. Many descriptions and mechanisms have been proposed for the stabilized and improved strength of the bonds in the presence of nanoparticles:

- According to the theory of the oxide layer breakdown, it can be argued that the greater the hardness of SiO₂ nanoparticles, as compared to that of aluminum, the further the breakdown of the surface oxide layer and, finally, the more the extrusion of pure metal.

- Shear deformation can result in the accumulation of dislocations around SiO₂ nanoparticles. Similar to the case of composite materials, this phenomenon leads to the local hardness increase in the interface of sheets and consequently, bond strength enhancement.

- Since the bond between particles and the metallic sheet is developed based on atomic diffusion mechanism, the maximum static friction and interface bond strength are the controlling parameters. The small size of particles is effective in the enhanced atomic diffusion of particles to the metallic sheet, leading to the enhanced bond strength increase and the improved strength.

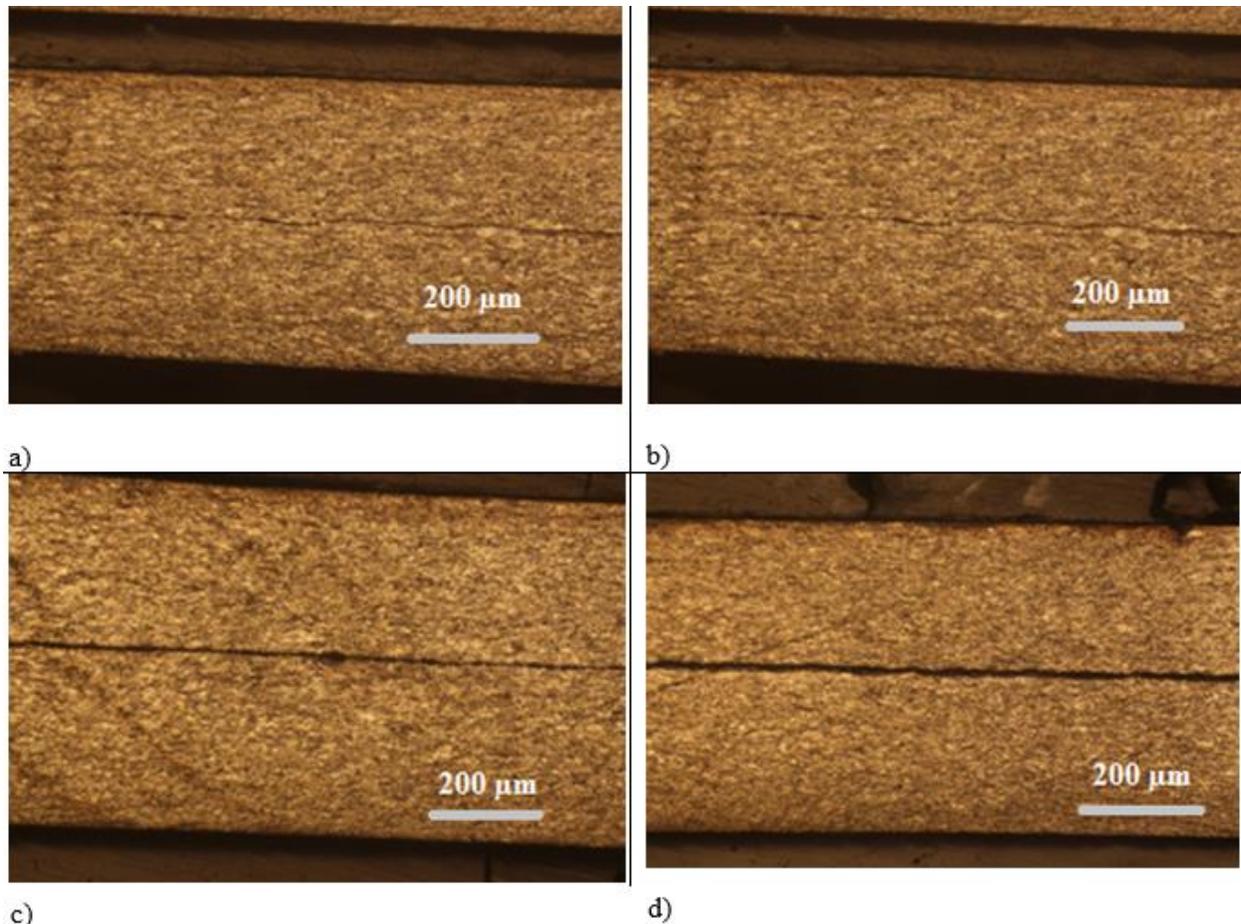


Fig. 7: Optical microscopy images of the RD-ND sheet of the cold rolled sheet: a) the monolithic CP-TiO₂; b) CP Ti/0.1wt.% nano-TiO₂; c) CP Ti/0.3wt.% nano-TiO₂; and d) CP Ti/0.5wt.% nano-TiO₂.

The mentioned points indicate the dual role of oxide layer failure mechanism. The presence of ceramic particles as the reinforcing phase because of its greater hardness as compared to the matrix, can facilitate oxide layer failure and leads to the further extrusion of the pure metal. On the other hand, the particles themselves might act as barriers against pure metals for reaching each other. The coincidence of these two effects ultimately results in either the improved or degraded bond strength between the two metals. The results of this work are consistent with those of the previous works. Figure 7 illustrates the optical microscope images of different specimens. As can be seen, the monolithic CP Ti sample exhibits a failure in the oxide hard layers as well as the bonds created on many sites. An increase in TiO₂ nanoparticle in CP Ti/0.1wt.% nanoTiO₂ sample was accompanied by a decrease

in the number of developed bonds, as compared to the monolithic specimen. By a further increase in the nanoparticles concentration, a decrease in the surface bonded areas was seen for CP Ti/0.5wt.% nanoTiO₂. The microscopic images showed a larger and more continuous area covered with nanoparticles, implying the barrier role of nanoparticles against the pure metal extruded from the interface of the surface oxide layers [18-20].

4-Conclusion

In the present work, the effect of the bond strength in commercially pure titanium (CP Ti) sheets processed by the accumulative roll bonding (ARB) was investigated. The main results could be outlined as follows:

1) The increase in TiO₂ nanoparticles content led to the declined peeling strength; the increase in

TiO₂ nanoparticle content from 0.1 to 0.5wt.% was accompanied by the reduced bond strength.

2) The presence of TiO₂ nanoparticles between CP Ti sheets caused the creation of a barrier that prevented observing the pure metal driven out of the interface of failure hard oxide layers and the development of suitable bonds between sheets, as manifested in the reduced strength measured through the peeling test.

3) As shown in the SEM images, the increase in TiO₂ nanoparticle concentration resulted in the creation of the non-unified expanded layers of these nanoparticles. Such layers could properly justify the reduced bond strength determined through the peeling test, as compared to the monolithic specimen and the reduced strength and the higher tolerance during the peeling test, particularly at the higher TiO₂ nanoparticle concentrations.

4) It seems that TiO₂ nanoparticle addition does not have a considerable effect on the further breakdown of surface oxide layers and the facilitated extrusion of the pure metal.

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