Microstructure and mechanical properties of the Ni/Ti/Nb multilayer composite manufactured by accumulative pack-roll bonding

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Abstract

In this work, the Ni/Ti/Nb multilayer composite was successfully manufactured by accumulative pack-roll bonding. The microstructure evolution and mechanical properties of the composite during the accumulative roll bonding (ARB) process were investigated by scanning electron microscopy(SEM), energy dispersive spectrometer(EDS), transmission electron microscopy (TEM), micro-hardness and tensile tests. The results showed that after 5 passes of the ARB process, the deformations of layers were relatively uniform, and no large number of interlayer fractures occurred. The microstructures of Ni and Ti were both equiaxed grains with a grain size of 200 nm and 150 nm, respectively, and finer equiaxed grains of the Ni layer were observed at the interface. The laminar structure of Nb layer was observed. The tensile strength and micro-hardness increased significantly as the number of ARB increased. After 5 passes of the ARB process, the tensile strength of the composite reached 792.3 MPa, and the micro-hardness of Ni, Ti, and Nb were increased to 270.2, 307.4, and 243.4 HV, respectively.

Keywords: Ni/Ti/Nb multilayer composite, accumulative roll bonding, microstructure, mechanical properties doi:10.20944/preprints202002.0030.v1

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1. Introduction

Metal multilayer composites generally consist of two or more different metals, which can combine the physical properties, chemical properties, and mechanical properties of different metals to improve the overall performance of composites [1-2]. Metal multilayer composites have been produced by some bonding methods: diffusion bonding [3], reaction bonding [4], explosive bonding [5], et al. Compared with several other methods, the roll bonding as the characteristics of simple process, low cost, effective and efficient production, has become the main method for manufacturing metal multilayer composites.

Accumulative roll bonding(ARB) is a kind of severe plastic deformation(SPD) technology, firstly proposed by Saito in 1998, which is a method for preparing ultrafine crystal plates [6]. Compared with other severe plastic deformation techniques, including: High pressure torsion(HPT), Equal channel angular pressing(ECAP), Repetitive corrugation and straightening(RCS), and Cyclic extrusion compression(CEC), ARB technology can not only prepare ultra-fine grain plates with excellent comprehensive properties, but also can produce multilayer composites with dissimilar materials [7-11]. In recent years, many scholars have successfully prepared multilayer metal composites by ARB process such as Al/Mg[12-14], Al/Ti[15-16], Al/Cu[17], Al/Ni[18-19], etc. Some scholars have also studied the three metal composites, such as Al/Cu/Sn [20], Al/Cu/Mn [21], Al/Cu/Ni [22], Al/Zn/Cu [23], Al/TI/Mg[24], etc. For composites consisting of three different metals, Al with low hardness and good deformability is usually used as the matrix, which is beneficial to the interface bonding in the ARB process. However，there are few researches on composites composed of three metals with high strength and hardness. Cold-roll bonding must have a large reduction to achieve interface bonding，but a large reduction is difficult for the metal with high hardness by cold-rolling. Therefore, the sample must be heated for roll-bonding, but it faces the problem of oxidation on the metal surface. These problems can be well solved by a pack-roll bonding process with an internal vacuum.

The pack-roll bonding mainly stacks the metal sheets into a pack and performs vacuum sealing treatment to prevent oxidation of the metal surfaces during the hot rolling process. It enhances interface bonding strength and improves the mechanical properties of composites.

Pure titanium is a hexagonal close-packed structure with low density, high specific strength, and strong corrosion resistance. Pure nickel is a face-centered cubic structure with high conductivity and thermal conductivity, strong toughness, and excellent plastic processing performance. Pure niobium has a high melting point, and adding niobium can improve the high-temperature performance of the composite.

In this study, commercial pure Ni, Ti, Nb are used as raw materials, and Ni/Ti/Nb multilayer composites are manufactured by accumulative pack-roll bonding. The microstructure evolution, the interface diffusion, and the mechanical properties have been studied by SEM, TEM, EDS, micro-hardness, and tensile tests.

2. Experimental procedures

In this work, commercial pure Ni, Ti, and Nb with a thickness of 1 mm were used as raw materials. The chemical compositions of Ni, Ti, and Nb are shown in Table 1.

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Table 1 Chemical composition of raw materials

	Element(wt%)								
	Nb	Ni	Ti	Cu	Si	Fe	Mn	Zn	C
Ni	-	Bal	-	0.002	0.01	0.01	0.03	-	0.011
Ti	-	-	Bal	0.01	0.4	0.3	-	0.04	0.01
Nb	Bal	-	-	-	0.001	0.001	0.03	-	0.003

The raw materials were cut into a dimension of 120 mm × 55 mm × 1 mm. The three kinds of metal sheets were degreased in acetone and polished with a diameter of 0.3 mm wire stainless steel brush. Two Ni, Ti sheets, and one Nb sheet were stacked neatly in the order of “Ni-Ti-Nb-Ti-Ni”. The raw materials were placed into a pack (The pack size is shown in Fig. 1(a).), and the pack was made of Q235 steel and pre-annealed at 800 °C. The pack was sealed by argon arc welding and vacuumed (as shown in Fig. 1(b)).

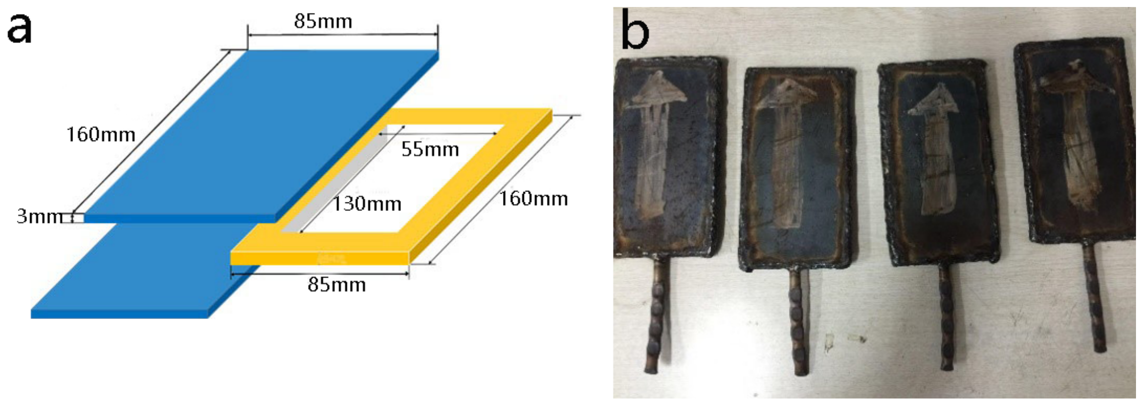

Fig. 1. Schematic of the pack: (a)the size of the pack, (b) pack was sealed and vacuumed.

The pack was heated in an electric resistance furnace for 10 minutes at 500 °C, and the non-lubricated rolling process was carried out with a total reduction of 66.7% (Von Mises equivalent strain ε=1.27). After the first pass, the bonded sheets were removed from the pack and cut into three by a shearing machine. The metal sheets were

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degreased in acetone again, and the surfaces were brushed with a steel brush and stacked on each other. The stacks were placed into a pack with sealed and vacuumed, and the rolling process was continued with a total reduction of 66.7%. The rolling process (as shown in Fig. 2) was repeated for up to 5 passes (Von Mises equivalent strain ε=6.35) carried out with no lubrication on a two-high mill with a roll diameter of 350 mm. The roll speed was set to 5 rpm during the ARB process.

The microstructure of the composite was observed by scanning electron microscopy (FEI Quanta 450 FEG) and transmission electron microscopy (TEM, Tecnai G2 F20). The diffusion data of each element at the interface of the composite material was collected by Energy Dispersive Spectrometer (EDS) to characterize the interface diffusion of samples. The Vickers micro-hardness (HV) of each layer of the composites was measured by micro-hardness tester (HXD-1000TM) at a load of 100 g for 15 s. Five points on each metal were measured for hardness and averaged. The tensile test was carried out using a WDW-200D microcomputer-controlled electronic universal material testing machine with a tensile speed of $1x10^{-3}$ s^{-1}. The tensile sample was processed in the rolling direction. The gauge length and width of thetensile samples were 10 mm and 3 mm.

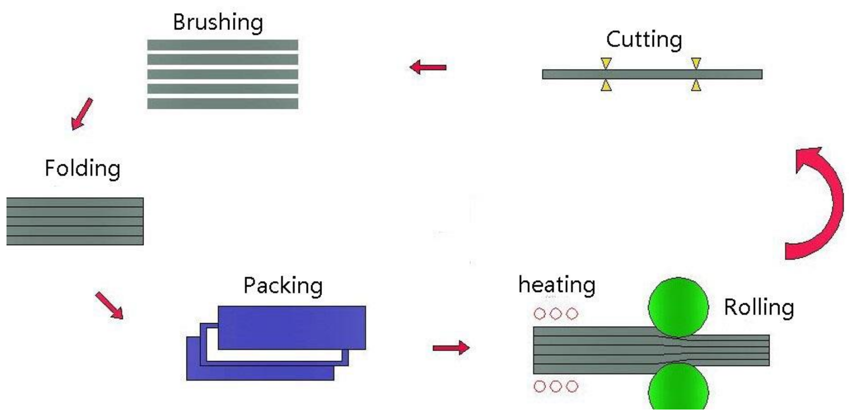

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Fig. 2. Schematic of the accumulative pack-roll bonding.

3. Result and discussion

3.1 Microstructure evolution

The microstructure of the Ni/Ti/Nb multilayer composites on the RD-ND plane during different ARB passes can be observed in Fig. 3. The three metals were uniformly distributed, and the thickness of layers was gradually reduced along the rolling direction. After 3 passes of the ARB process, the interfaces of layers were relatively straight, and no obvious necking and fracturing appeared. After the fourth pass, severe necking occurred, and some Nb layers fractured. Some shear bands at an angle of about 45° to the rolling direction appeared after 5 passes of the ARB process. Similar results were found in other studies on metal composites [17-19, 24-27]. These studies had indicated that relatively hard metals were prone to local necking and fracturing during the ARB process. As the number of ARB passes increased, hard metals were separated and evenly distributed in soft layers. It was because as the number of ARB passes increased, the shear force penetrated from the surface to the inside of metal layers, and a distinct shear band appeared. Besides, due to the different plastic deformation of dissimilar metals, when the accumulative strain reached a certain level, uneven deformation occurred locally in layers to cause a change in stress state, and the stress reached or exceeded the yield limit of the material, resulting in necking and fracturing in harder metal.

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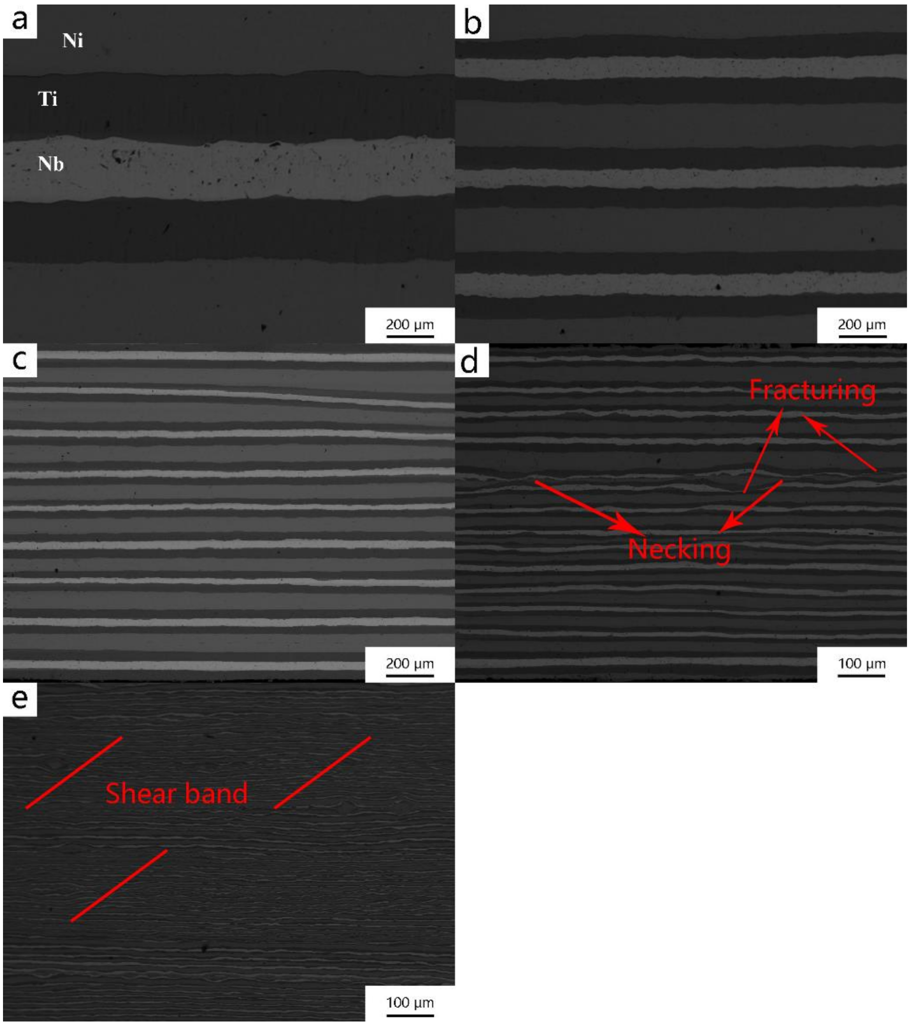

Fig. 3. SEM micrographs of the Ni/Ti/Nb composite: (a)after the first pass, (b)after the second pass, (c)after the third pass, (d)after the fourth pass, (e)after the fifth pass.

Fig. 4 is a TEM micrograph of the composite after different ARB passes. The microstructure of the Ni layer after the 3rd and 5th passes are shown in Fig. 4(a) and 4(b), respectively. It could be observed that as the number of the ARB passes increased, the grain size of Ni was significantly reduced, and fine equiaxed grains with a size of about 200 nm appeared after the 5 passes. The Ni layer had a face-centered cubic structure, and the dislocation slip caused the dislocation density inside the grain and at the grain boundary to increase rapidly, and the interaction between the dislocations was plugged and entangled to form dislocation cells. Then the dislocation cells evolved into grain boundaries, forming fine grains. The refinement mechanism was that the

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dislocation interfaces are formed, and the grains were divided continuously.

In addition, it was found in Fig. 5(b) that there were some finer equiaxed grains near the interface, and the dislocation density was larger than that away from the interface. During the ARB process, due to the different flow rates of dissimilar metals, severe shear strain was generated near the interface of the sample, resulting in grain refinement and large dislocation density.

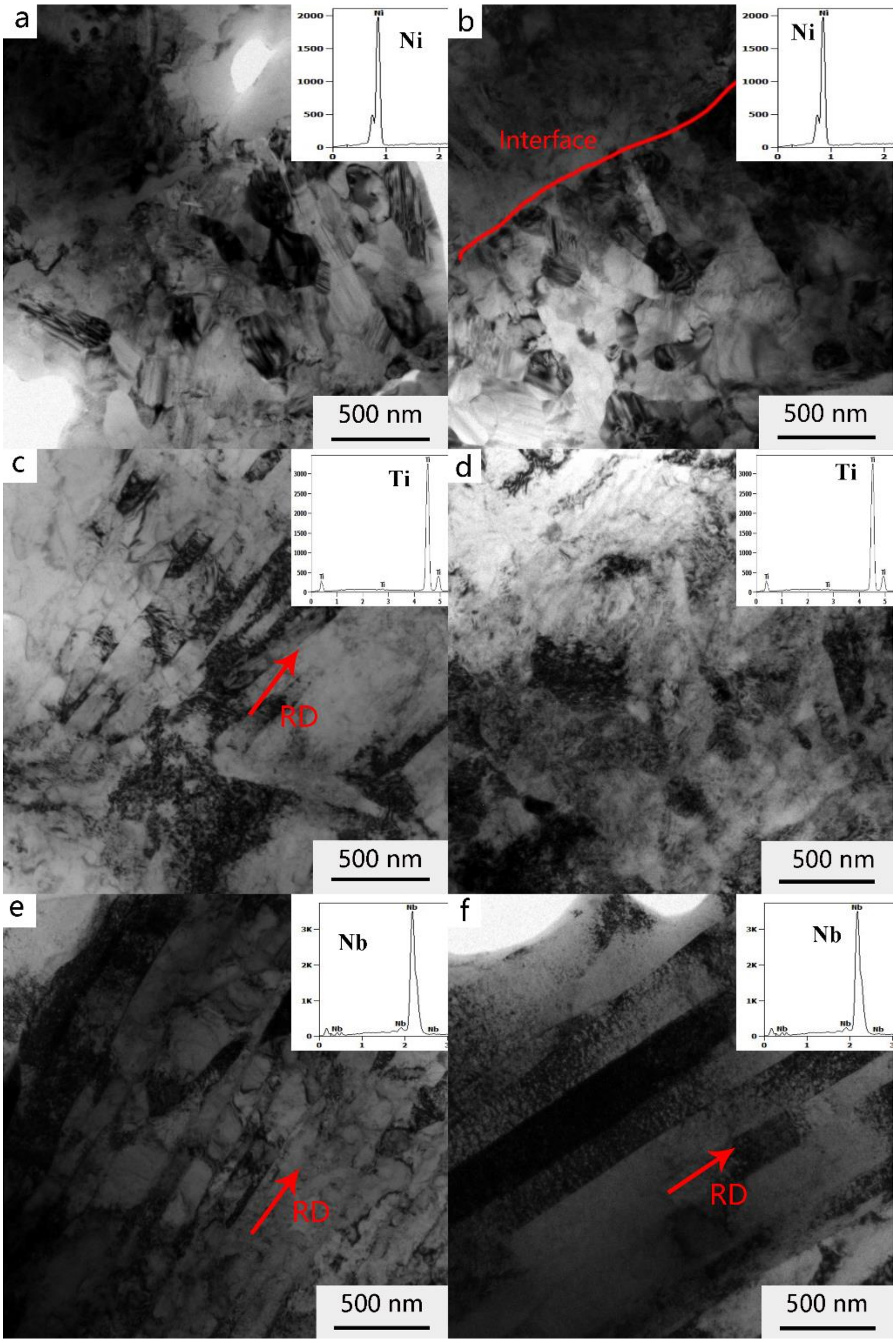

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Fig. 4. TEM micrographs of the Ni/Ti/Nb composite: (a)Ni layer after third pass, (b)Ni layer after the fifth pass, (c)Ti layer after the third pass, (d)Ti layer after the fifth, (e)Nb layer after the third pass, (f)Nb layer after the fifth pass.

The microstructure of the Ti layer after the 3rd and 5th passes are shown in Fig. 4(c) and 4(d), respectively. It could be seen that, after the third pass of the ARB process, the Ti layer grains were flattened and elongated in the rolling direction, forming a laminar structure, and there was a large density dislocation between the laths. With the increasing number of the ARB passes, the grains were elongated and fractured in the rolling direction, and dynamic recovery and recrystallization occurred. After 5 passes, fine equiaxed grains appeared with an average grain size of about 150 nm.

The Ti layer had fewer slip systems due to the close-packed hexagonal structure. In the initial stage of plastic deformation, twin deformation was easy to occur, and the dislocations slipping on the base surface were transferred to the prism surface by the slip and decomposed. And then the slip on the prism surface played a leading role. As the deformation continues, the dislocations continued to proliferate, move, and interact to form dislocation walls. The dislocation walls divided the large-size grains into several sub-grains, forming dislocation cells. When the dislocation density of the cell boundary reached a certain level, the dislocations generated by the deformation were absorbed at the grain boundary, the dislocation proliferation and annihilation were balanced, and the grain orientation difference was further increased. At the same time, dynamic recovery and recrystallization occurred during the deformation process, resulting in more fine equiaxed grains.

The microstructure of the Nb layer after the 3rd and 5th passes are shown in Fig. 4(e) and 4(f), respectively. The laminar structure of Nb layer was observed, and the grains were oriented parallel to the rolling direction. From the third pass to the fifth pass, the lath width was reduced, and the dislocation density was increased. This phenomenon was due to the high recrystallization temperature of Nb, and the ARB process at 500 °C was insufficient to provide the energy required for dynamic recrystallization. Therefore, as the strain accumulated, the width of the lath decreased, and a large number of dislocations appeared inside the grain and at the grain boundaries.

3.2 Interface investigation

In the initial stage of ARB, the interface bonding method is mainly mechanical bonding, that is, under the action of interlayer friction, stress concentration and cracking occur locally on the relatively hard metal surface, forming a crack perpendicular to the rolling direction. The relatively soft metal is squeezed into the surface layer of the hard layer through the cracks under the rolling pressure, and then in the process of plastic deformation, the interfaces are compressed and rotated due to the combined action of compressive stress and shear stress, forming a firm mechanical combination.

As the ARB process, elemental diffusion occurs at the interface, and the interface bonding mode is similar to that of powder metallurgy (mechanical alloying). Mechanical alloying involves three main mechanisms: (1) atomic displacement due to mechanical force, (2) diffusion around vacancies, and (3) produced vacancies as a result of plastic deformation [18,19,24].

Fig 5 shows the EDS analysis of the interface line after different passes. The elemental diffusion distance of Ni/Ti and Nb/Ti interfaces after different ARB passes is illustrated in Fig. 5(d). It could be seen that as the ARB process, the element diffusion

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distance increased, and the increasing rate of the diffusion distance increased, which might be related to the preheating before rolling. Besides, it could be found that the element diffusion distance of the Ni/Ti interface was always larger than that of the Nb/Ti interface, indicating that in the same conditions, the interface diffusion distance was related to the nature of the elements. Nb has a higher energy level due to its larger atomic number, and atomic diffusion requires more energy. On the other hand, the melting point of Nb is much higher than that of Ni, which causes the slow diffusion of Nb. As the ARB process, the element diffusion distance between the interfaces increased, indicating that the interface bonding strength was enhanced. Metal bonds were formed due to the balance of attractive and repulsive forces and the reduction in interface energy. This interface diffusion due to deformation has a positive effect on improving the mechanical properties of the composite.

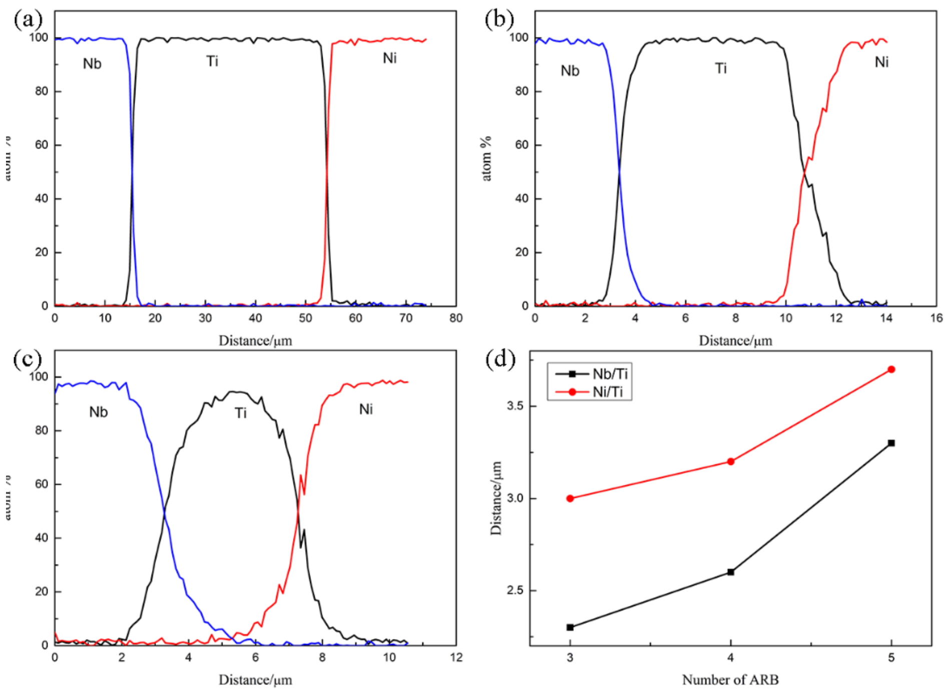

Fig. 5. The interface diffusion distance of the Ni/Ti/Nb composite: (a)after the third pass, (b)after the fourth pass, (c)after the fifth pass, (d)variations of diffusion distance versus the number of ARB pass.

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3.3 Mechanical properties

3.3.1 Tensile test

In order to study the effect of ARB cycle on the mechanical properties of composites, a tensile test was conducted. Figure 6(a) shows the stress-strain curves of the composite after different passes. Figure 6(b) shows the change in the mechanical properties of the composite with different ARB passes. It could be found that after the first pass rolling, the strength of the composite increased significantly, and the elongation decreased compared with the raw materials. As the number of ARB passes increased, the strength of the composite gradually increased, and the elongation decreased. The tensile strength of the composite material reached 792.3 MPa after the fifth pass.

In general, there were two main strengthening mechanisms for the improvement of composite strength: work hardening and grain refinement. In the initial stage of ARB, strain hardening or dislocation strengthening played a major role in the improvement of composite strength. With the increasing number of ARB passes, the microstructure of the composite was gradually refined and uniform, and dynamic recovery and recrystallization occurred. The higher strength of the composite was related to grain refinement (ultrafine grains played a major role in strengthening) [24,28]. Additionally, due to the close mechanical properties of the three metals, the deformation of each layer was relatively uniform during the ARB process, and inter-laminar fractures rarely occurred. Due to the increased interface, a large number of coherent and evenly distributed interfaces hindered the slip and expansion of dislocations. The strength of the composites had been increased due to the dynamic recovery and recrystallization, grain refinement.

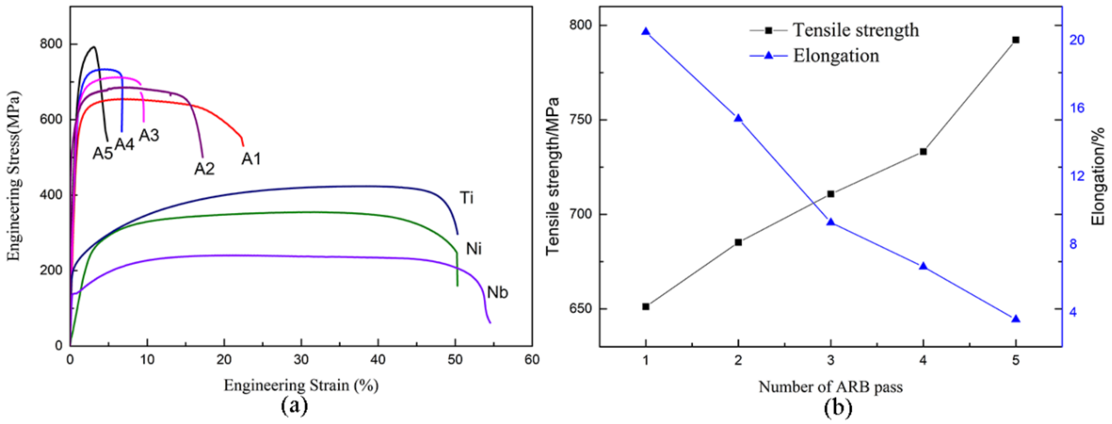

Fig. 6. Mechanical properties of Ni/Ti/Nb multilayer composite: (a) stress-strain curves, (b) variations of tensile strength and elongation versus the number of ARB passes.

3.3.2 Micro-hardness

The variation of average micro-hardness with respect to the number of ARB pass is shown in Fig. 7. As the number of ARB passes increased, the average micro-hardness of layers increased gradually due to the work hardening and grain strengthening. After the first pass, the micro-hardness of the three metals increased rapidly, and in the subsequent process, the rate of increase decreased. It was reported that [18], the work hardening affected in the initial stage of the ARB process, the effect of improving the micro-hardness was much higher than the later grain refinement. Due to the different mechanical properties and flow characteristics of the three metals, the strain was uniformly allocated between the soft phase and the hard phase during the deformation process, resulting in different actual strains applied to the three metals, the hardness difference of the three metals was gradually reduced. After 5 passes, the average micro-hardness of Ni, Ti, Nb increased from 161.8, 173.4, and 78.2 HV to 270.2, 307.4, and 243.4 HV, which increased by 67.0%, 77.3%, and 211.3%, respectively. In the ARB process, the Nb grain was always laminar structure throughout the process, and no dynamic recovery and recrystallization occurred, so the hardness growth rate of Nb was

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the largest. The hardness increase rate of Ti was greater than Ni because of its HCP structure and the high work hardening rate.

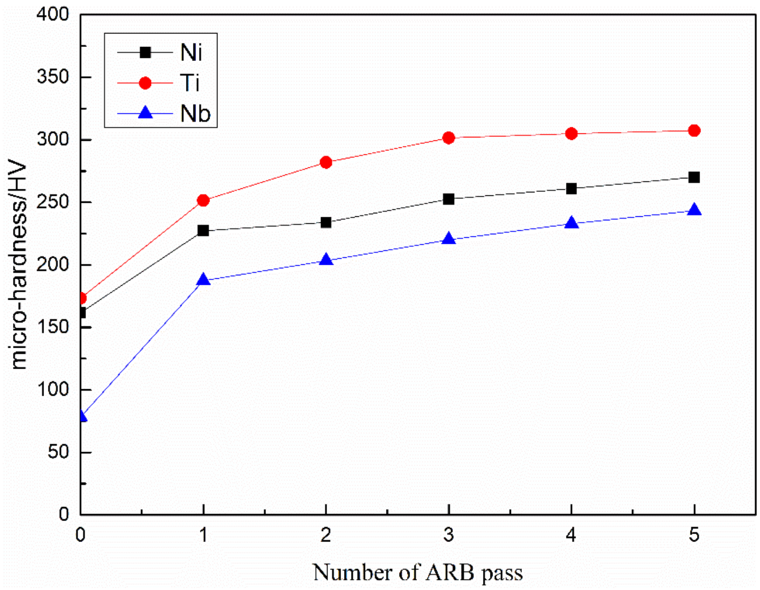

Fig. 7. variations of micro-hardness versus the number of ARB passes

3.3.3 Fractography

Fig. 8 shows the fracture surface of the composite after tensile testing. It could be seen that after the third pass, there was obvious interface separation in the fracture surface, and after the fifth pass, the interface was well combined. It was indicated that as the ARB process, the interfacial bonding strength of the composite was enhanced gradually. Fig. 8(c) and 8(d) show the fracture surface of the composite at higher magnifications. It could be seen that after the third pass, Nb and Ti were nearly integrated, and there was no dimple on the fracture surface. It was exhibited a plastic slip fracture on the surface. There were obvious dimples on the fracture surface of the Ni layer, which was the dimple fracture. This phenomenon was related to the microstructure of the three metals (as shown in Fig. 5). After the 5th pass, the composite became a whole due to the enhanced interface bonding. The dimples on the fracture surface were not as deep

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as that in the third pass, and the equiaxed dimples became shallow and small shear dimples due to shear stress. Therefore, the fracture surface exhibited a shear fracture.

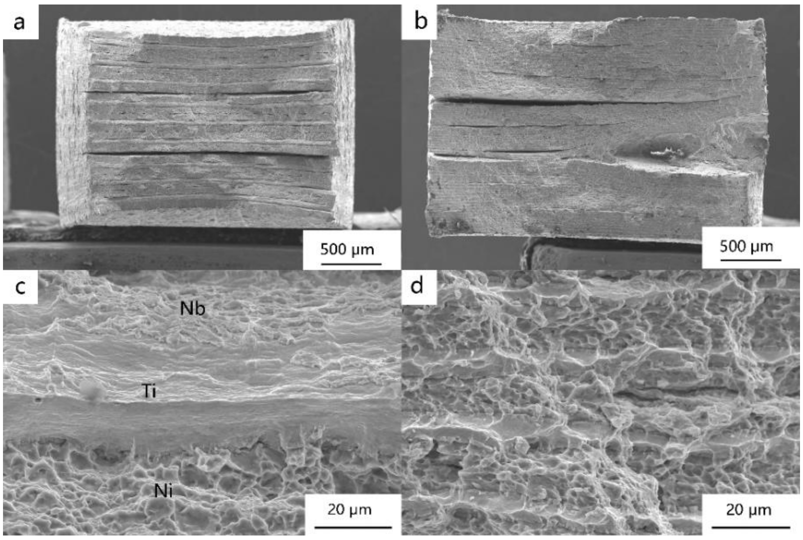

Fig. 8. Tensile fracture surfaces of the Ni/Ti/Nb multilayer composite: (a)after the third pass, (b)after the fifth pass, (c)high magnification after the third pass, (d) high magnification after the fifth pass.

4. Conclusions

In this study, the Ni/Ti/Nb multilayer composite was manufactured by accumulative pack-roll bonding. The microstructure evolution, interface diffusion, and mechanical properties were investigated. The results can be listed as follows:

(1) The Ni/Ti/Nb multilayer composite was manufactured by ARB technology up to 5 passes. The deformation of each layer was relatively uniform.

(2) As the number of ARB process increased, the grain size of the composite decreased. After 5 passes, the microstructures of Ni and Ti were equiaxed grains with the grain size of 200 nm and 150 nm, respectively, and finer equiaxed grains were observed at the interface of the Ni layer. The laminar structure of Nb layer was observed. The Nb grains were oriented parallel to the rolling direction.

(3) As the number of ARB process increased, the tensile strength of the composite increased significantly, and the elongation decreased. After 5 passes, the tensile

strength of the composite reached 792.3 MPa.

(4) After 5 passes, the micro-hardness of Ni, Ti, and Nb were increased to 270.2, 307.4, and 243.4 HV, and the micro-hardness difference among the three metals was gradually decreased.

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