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# Strengthening a multilayered Zr/Ti composite by quenching at higher temperature

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ABSTRACT

By rolling and diffusion bonding, multilayered Zr/Ti composites were fabricated using commercial pure titanium (CP-Ti) and zirconium (Zr702) sheet as raw materials. For the as-fabricated Zr/Ti laminated metal composites (LMCs), quenching at different temperatures were carried out to refine the microstructure. Microstructures of Zr/Ti LMCs before and after the quenching were characterized by scanning electron microscope and electron backscatter diffraction. Micro-hardness and compression tests were performed to reveal the effect of quenching temperature on the mechanical properties of the Zr/Ti LMCs. It indicates that certain part of the microstructure can be refined by phase transition during quenching. The volume fraction of the quenching-affected (refined) microstructure increase with the increase of quenching temperature. Micro-hardness tests show that the hardness is not uniform along the thickness of the Zr/Ti LMCs due to the heterogeneous chemical composition and microstructure. The compressive strength of the Zr/Ti LMCs increases with the increase of the quenching temperature, which is mainly attributed to the fact that more microstructures have been refined in higher temperature quenching.

## 1. Introduction

Zirconium (Zr) alloys, such as Zr-Sn [1–3], Zr-Nb [4,5] and Zr-Sn-Nb [6,7], are extensively used in the nuclear industry as cladding materials because of their high corrosion resistance, small capture cross section for thermal neutrons and excellent compatibility with nuclear fuels [8–10]. However, traditional Zr alloys, which have been used in the nuclear industry, normally with low strength, can't meet the requirements of structural materials in many other fields, such as aerospace and aviation. New Zr alloys such as ZrB [11,12], ZrTi [13–15], ZrTiAlV [16] and ZrTiMoSn [17] have been developed to explore the application of Zr alloys as structural materials in industries other than the nuclear industry.

Titanium (Ti) and Zr are the same group elements in the periodic table of the chemical elements and show similar performances. At high temperature ($>$ 882 °C for Ti, $>$ 863 °C for Zr), the stable phase is β phase, which has a body-centered-cubic (BCC) structure. While, at room temperature, the stable phase is α phase, which has a hexagonal-closed-packed (HCP) structure. The Zr-Ti system was reported to achieve a complete solid solution in the equilibrium phase state for both high and low temperatures [18]. For Zr-Ti binary alloys, due to solid solution strengthening and grain refinement, the tensile strength increase with the increase of Ti ($<$ 50 at%) concentration. However, the increase in strength is generally accompanied by the reduction of ductility in traditional metallic materials [19–21]. A maximum tensile strength of ~1170 MPa can be achieved in Zr-Ti binary alloys, which only has a elongation of ~5% [22].

Laminated metal composites (LMCs) have attracted attention due to their mechanical properties [23,24] that cannot be obtained from a single material. Recently, many researches have been conducted on LMCs aiming to combine high strength and high ductility [25–27]. The layered structure in LMCs can provide extra work hardening capability and cause multiple deflection of the propagation path of micro-cracks, thus generally leading to a higher ductility [28,29]. In our previous work [30], intermetallic-compound-free Zr/Ti laminated metal composites (Zr/Ti LMCs) were fabricated by diffusion bonding. The width of the mutual diffusion region increased with the annealing temperature and holding time [30]. Meanwhile, the compressive yield strength of Zr/Ti LMCs increased with the growing of the mutual diffusion region as results of the solid solution strengthening [30]. The strength of Zr/Ti LMCs may be further improved by tailoring the microstructures, for example refining the microstructure, since very coarse grains (many grains having size $>$ 300 μm) were obtained in our previous work [30].

In this work, quenching process was adopted to tailor the microstructure of Zr/Ti LMCs. The microstructures before and after quenching were carefully characterized by scanning electron

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**Table 1**
Nominal chemical compositions of Zr702 and CP-Ti.

| Material | Hf | Sn | Fe + Cr | Zr | |
|---|---|---|---|---|---|
| **Zr702** | 0.95 | 0.16 | 0.15 | Balance | – |
| **Material** | **Fe** | **C** | **N** | **O** | **Ti** |
| **CP-Ti** | 0.04 | 0.01 | 0.003 | 0.04 | Balance |

microscopy (SEM) and electron backscatter diffraction (EBSD) to reveal the effect of quenching treatment on the mechanical properties of Zr/Ti LMCs. As a result of the gradient distribution of the chemical composition, quenching at different temperatures cause the refinement of different volume fractions of microstructure, thus significantly affecting the mechanical property of Zr/Ti LMCs.

## 2. Experimental procedures

### *2.1. Materials*

The raw materials used in this study were commercial pure zirconium (Zr702) and commercial pure titanium (CP-Ti) rolled sheet with thickness of 0.5 mm. The nominal chemical compositions of the as-received Zr702 and CP-Ti sheets are listed in Table 1. The starting microstructures of the CP-Ti sheet and Zr702 sheet, which were characterized by electron backscatter diffraction (EBSD), are presented in Fig. 1. It is revealed that the initial microstructures of Zr702 and CP-Ti consist of recrystallized equiaxed grains. The average grain sizes for the raw Zr702 and CP-Ti sheets are ~ 14 μm and ~ 32 μm. All average grain sizes presented in current study are measured by linear intercept method by using commercial available Channel 5 package.

### *2.2. Diffusion bonding process*

Cold rolling and diffusion annealing were used to fabricate Zr/Ti LMCs in this work. A group of small sheets, with size of 100 mm (length, along the rolling direction, RD) × 20 mm (width, along the transverse direction, TD) × 0.5 mm (thickness, along the normal direction, ND), were cut from the raw Zr702 and CP-Ti sheets by wire electrode cutting. To remove the oxidization layers of the Zr702 and CP-Ti sheets, pickling process was carried out at room temperature. Surface-cleaned Zr702 sheets and CP-Ti sheets were stacked alternately, as illustrated in Fig. 2. Then, cold rolling (~60% reduction in one pass) and vacuum diffusion bonding annealing were conducted to fabricate the Zr/Ti LMCs. The vacuum diffusion bonding annealing was performed at 800 °C and hold for 7 h. During vacuum diffusion bonding, the Zr/Ti billets were always fixed between two steel plates. Pressure was provided by the screws that connecting the two steel plates during diffusion bonding. After the diffusion bonding annealing, it was slowly cooled down in furnace. For comparison, Zr/Zr LMCs and Ti/Ti LMCs were also fabricated using same processing as for the Zr/Ti LMCs.

### *2.3. Quenching process*

For quenching, specimens (6 mm × 5 mm × 2 mm) were cut from the fabricated Zr/Ti LMCs. The samples were heated up to 650 °C, 700 °C, 750 °C, 800 °C, 900 °C and holding for 30 min, which were followed by quenching into cold water (~ 25 °C). During the heating and heat preservation process, Zr/Ti LMCs samples were sealed in a vacuum quartz tube to avoid oxidation. In the following text, the quenched samples are named after the quenching temperature as 650Q, 700Q, 750Q, 750Q, 800Q and 900Q. For comparison, Zr/Zr LMCs and Ti/Ti LMCs were also quenched at 900 °C and denoted as Zr/Zr-900Q and Ti/Ti-900Q, respectively.

### *2.4. Mechanical properties and microstructure*

For mechanical properties tests, samples with size of 6 mm (RD) × 5 mm (TD) × (ND) 2 mm were cut from LMCs sheets. The room temperature compressive properties of LMCs samples were measured using an AG-X50kN machine with a fixed stretching speed of 0.5 mm/min. To avoid anisotropy issue, all compressions were along RD. Compression test for each sample was repeated three times for the reliability and repeatability of the experimental data. On the cross-section (RD-ND plane) of Zr/Ti LMCs, Vicker's micro-hardness tests were carried out. A 500 g force was applied, and the load was held for 15 s in the micro-hardness tests. 36 indentations at least were performed for each sample.

Microstructures and chemical composition distributions of the Zr/Ti LMCs were investigated by scanning electron microscopy (TESCAN MIRA3), electron backscatter diffraction (EBSD) and energy dispersive spectroscopy (EDS). The region for microstructural characterization was located at the center of RD-ND plane. EBSD data for the as-fabricated and the quenched samples were collected using a step size of 1.5 and 0.3 μm, respectively.

## 3. Results and discussions

### *3.1. As-fabricated microstructures*

Typical macrostructure of the as-fabricated Zr/Ti LMCs is shown in Fig. 3(a). EDS line scanning results are also presented in Fig. 3(a). It

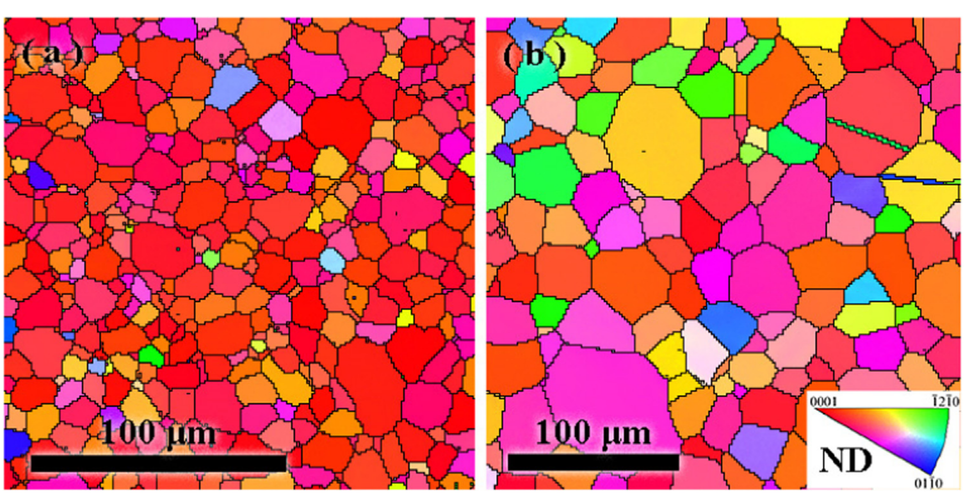


**Fig. 1.** Starting microstructures of the raw (a) Zr702 and (b) CP-Ti sheets.