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ORIGINAL REPORT



Fabrication of multi-element alloys by twin wire arc additive manufacturing combined with in-situ alloying

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ABSTRACT

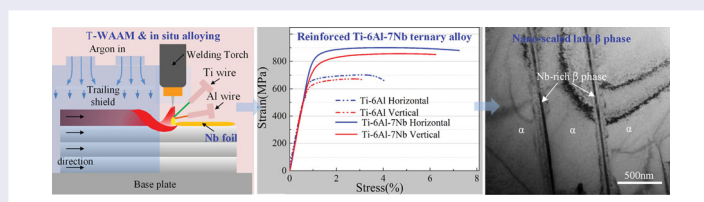
In this work, twin wire arc additive manufacturing (T-WAAM) combined with in-situ alloying method was developed to fabricate multi-element alloys, and biomedical Ti-6Al-7Nb alloy with a good combination of strength and ductility was successfully fabricated for the first time. The modification of microstructure and the improvement of mechanical properties of the deposited Ti-6Al-7Nb alloy caused by Nb addition were investigated. Although demonstrated here for Ti-6Al-7Nb alloy, the combined method of T-WAAM and in-situ alloying is in principle applicable to fabricate other multi-element alloys by composition design, which is conducive to expanding the application fields of WAAM.

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KEYWORDS

Wire arc additive manufacturing; in-situ alloying; titanium alloy



IMPACT STATEMENT

Twin wire arc additive manufacturing combined with in-situ alloying was developed to fabricate multi-element alloys, and biomedical Ti-6Al-7Nb alloy with a good combination of strength and ductility was first fabricated.

Wire arc additive manufacturing (WAAM), characterized by a high deposition rate and low equipment costs, is attracting increasing interest on the fabrication of large-sized metallic parts though the precision of the as-built parts may be lower than those obtained using powder-bed systems [1–4]. This technology utilizes metal wire as the feedstock, electric arc as the heat source, and manipulation is done with either a robot or numerical control system [5,6]. Consequently, it is conceivable that the composition of deposited alloy is dependent on the types of metal wire fed into the moving molten pool. Unfortunately, commercially available metal wires are limited, particularly, some metal wires are difficult to manufacture since their nature brittleness and high cost, which seriously limits the design of alloy composition during WAAM.

To solve this problem, twin wire arc additive manufacturing (T-WAAM) has been proposed to fabricate alloys with variable compositions by simply adjusting the relative feeding speed of the two different wires [7]. By this approach, Ti-Al, Fe-Al, Ti-Ni and Al-Cu-Mg alloys were successfully deposited from the combination of commercial metal wires [8–11]. Although T-WAAM is a big step forward, it is still a challenge to fabricate multi-element alloys with a variable composition. For example, the WAAM of Ti-6Al-7Nb ternary alloy, which is a new generation of surgical implants and exhibits excellent biocompatibility, superior corrosion resistance and high specific strength [12–14], has been not realized until now. Based on T-WAAM, the introduction of other components is a facile way to fabricate multi-element alloys. In-situ alloying, which is a common method used

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to tailor the microstructure and improve the mechanical properties of the WAAMed titanium alloys [15–20], provides a simple way to add alloy elements into the deposited alloy. Hence, in principle, the fabrication of multi-element alloys could be realized by the combination of T-WAAM and in-situ alloying.

In this work, a combined method of T-WAAM and in-situ alloying was developed to fabricate a ternary alloy of Ti-6Al-7Nb for the first time. By T-WAAM, Ti-6Al alloy could be deposited by simply adjusting the relative feeding speed of Ti and Al metal wires; the alloyed element of Nb was added simultaneously into the molten pool of Ti-6Al in the form of foil. To highlight the effect of in-situ alloying of Nb, binary Ti-6Al alloy fabricated by T-WAAM was used as a comparative sample. The chemical composition, microstructure and mechanical properties of the deposited alloys were investigated, and the microstructural evolution mechanism caused by multiple thermal cycles during T-WAAM process was discussed.

Gas tungsten arc welding (GTAW) was used as the heat source. Pure Ti and Al wires with a diameter of 1.2 mm were used as the feedstock. Each deposition layer was formed by torch moving in a linear direction while the wires fed into the molten pool. The interlayer temperature measured by an infrared thermometer was fixed at 80°C. When a deposition layer finished, the height of torch increased approximately 1.2 mm to create subsequent layer. Nb foil with 50 μm in thickness and 11 mm in width was pre-clamped at the two ends to keep a close contact with the previous deposition layer. During T-WAAM process, the Nb foil melted into the molten pool together with Ti and Al wires. The main deposition parameters are listed in Table 1. It is worth noting that the composition of the ternary Ti-Al-Nb alloy could be adjusted by controlling the relative feeding speed of Ti and Al metal wires as well as the dimension of Nb foil. The content of Nb added into each deposition layer was controlled by adjusting its width, and the designed composition of the deposited alloy was Ti-6Al-7Nb. It was reported that the interior of the moving molten pool was dynamic due to the vigorous stirring under the action of arc [21]. Therefore, the composition was quite uniform and there is no obvious macro segregation of Nb in the deposition alloy as proved by the following EDS analysis. To prevent the alloy from oxidation, an inert trailing shield was applied behind the tail of the welding torch. The schematic of the in-situ alloying assisted T-WAAM process is shown in Figure 1(a) and the images of the T-WAAM setup are given in Figure S1.

The microstructure of the deposited Ti-6Al and Ti-6Al-7Nb alloys was observed by a Zeiss Smartzoom5 optical microscope (OM). Phase structure of the two deposited alloys was detected by X-ray diffractometer

Table 1. Deposition parameters of T-WAAM.

Deposition parameter	Value
Flow rate of argon (L/min)	10 (welding torch) 20 (trailing shield)
Current (A)	120
Welding speed (mm/s)	2
Electrode to workpiece (mm)	3
Wire feeding speed (cm/min)	Ti = 170, Al = 18 (Ti-6Al) Ti = 156, Al = 18 (Ti-6Al-7Nb)
Interlayer temperature (°C)	80

(XRD, Bruker D8 Advanced) using Cu Ka ($\lambda = 15,418 \text{ \AA}$) irradiation. Tensile specimens with a gage length of 60 mm and a $2.6 \times 1.0 \text{ mm}^2$ cross-section within the gage length were fetched from both deposition (horizontal sample) and build-up (vertical sample) directions of the deposited alloy. Five tensile specimens were tested using micro mechanical tensile test machine (Instron Micro Tester 5848) with a constant tensile velocity of 0.25 mm/min. The cutting position and quantity of tensile and microstructure characterization specimens are shown in Figure S2. The microstructure and chemical composition of the deposited alloy and the fracture morphology after tensile test were analyzed using SEM (JSM-7800F) equipped with an energy dispersive spectroscope (EDS). The β phase was observed by a transmission electron microscope (TEM, JEM-2100F) coupled with a selected area electron diffractometer (SAED). TEM sample was prepared by a focused ion beam (FIB).

Figure 1(b) shows the appearance of Ti-6Al-7Nb alloy produced by in-situ alloying assisted T-WAAM. The newly deposited layers exhibited bright metallic luster due to the application of inert trailing shield. Furthermore, the oxygen and nitrogen contents of the deposited alloy measured by Leco combustion instrument were 0.09 and 0.01 (wt.%), respectively, proving that the deposited titanium alloy was not oxidized and the protective effect of the inert trailing shield was satisfactory. The XRD results in Figure 1(c) indicated that α phase with a closely packed hexagonal lattice was the dominant phase for both the deposited Ti-6Al and Ti-6Al-7Nb alloys. Moreover, the diffraction peaks of Ti-6Al-7Nb alloy at $33\text{--}41^\circ$ were broaden, suggesting that the grain refinement was obtained by adding Nb (also proved by EBSD analysis in Figure S3). Figure 1(d) depicts typical stress–strain curves of the two deposited alloys both in horizontal and vertical directions. The measurement of ultimate tensile strength (UTS) and elongation (EL) reflected that the mechanical properties of the Ti-6Al-7Nb alloy were superior to Ti-6Al regardless of the tensile direction, demonstrating the positive role of Nb. The average UTS of deposited Ti-6Al-7Nb alloy in horizontal direction was $910 \pm 15 \text{ MPa}$ in combination with good ductility of $7.2\% \pm 0.5\%$, which increased by 35%

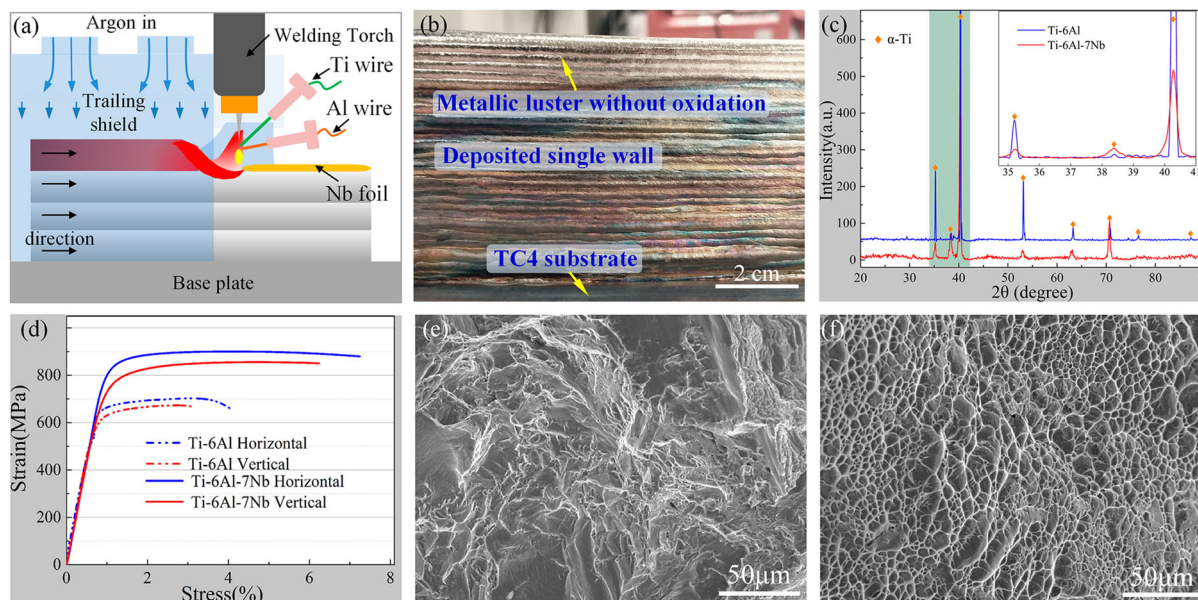


Figure 1. Characteristic of the deposited alloys fabricated by in-situ alloying assisted T-WAAM: (a) schematic of T-WAAM combined with in-situ alloying process; (b) single wall photo of Ti-6Al-7Nb alloy; (c) XRD patterns; (d) typical stress-strain curves at different directions; and typical fracture morphologies of (e) Ti-6Al and (f) Ti-6Al-7Nb.

and 56%, respectively, compared to Ti-6Al. Moreover, the anisotropy of mechanical properties, which has been widely reported in the AM of titanium alloys, could be also found for both of the two deposited alloys. The UTS and EL were 875 ± 18 MPa and $6.2\% \pm 0.4\%$ for Ti-6Al-7Nb alloy in vertical direction, respectively. The increment of UTS and EL in vertical direction reached 36% and 47% though the existence of anisotropy.

Figure 1(e,f) shows the fracture morphologies of the vertical samples of the two deposited alloys. The fractograph of Ti-6Al alloy exhibited typical brittle fracture characteristics since the appearance of dominant cleavage planes. Although some dimples could be observed on the fracture surface, the number of dimples was few. A ductile-like fracture surface was clearly observed for Ti-6Al-7Nb, as evidenced by the existence of dimples, which is in excellent agreement with the large elongation. Additionally, there are no signs of cleavage on the fracture surface.

Figure 2(a) shows the cross-section microstructure of the deposited Ti-6Al alloy observed by OM. Horizontal light-colored band that appeared periodically except for the last four deposition layers could be clearly observed. This band, which has been widely reported in the WAAM of titanium alloys, was named as HAZ band [22]. As seen in Figure 2(b), the microstructure of non-HAZ region was mainly composed of α phase with the grain size of 200–500 μm , which is similar to that of WAAMed pure titanium [23]. There is no significant difference of HAZ bands as seen in the magnified microstructure showing

in Figure 2(c), indicating the whole microstructure of deposited Ti-6Al alloy was uniform.

The feature of HAZ bands in deposited Ti-6Al-7Nb alloy was more distinct due to the addition of alloying element of Nb. From Figure 2(d), periodic HAZ bands with a width of 400–500 μm appeared. Similarly, it disappeared in the last four deposition layers. By comparing Figure 2(e,f), typical Widmannstatten structure with basket weave characteristics formed in the whole deposited alloy, and the microstructure in the HAZ band was finer than that in non-HAZ region because of the special thermal cycling experience. It is worth noting that the defects of micro-voids and cracks did not form in the deposited alloy fabricated by T-WAAM combined with in-situ alloying [24,25].

The characteristic of HAZ band was further observed by SEM. As seen in Figure 3(a), the microstructure of HAZ band was brighter and finer than that of non-HAZ region. The measured width of α sheet at the HAZ band was approximately 1 μm while that at the non-HAZ zone reached 5 μm . The EDS line scanning across a complete HAZ band of the deposited Ti-6Al-7Nb alloy is depicted in Figure 3(b). The composition of primary elements at different zones did not fluctuate significantly, except for 7.29% Nb content in HAZ band was slightly higher than that in the non-HAZ region (6.56 wt.%). Besides, the EDS composition distribution of the deposited alloy is quite uniform in the other two directions. It is obvious that the proportion of β phase had a close relationship with the Nb content because Nb was a β phase stabilizing

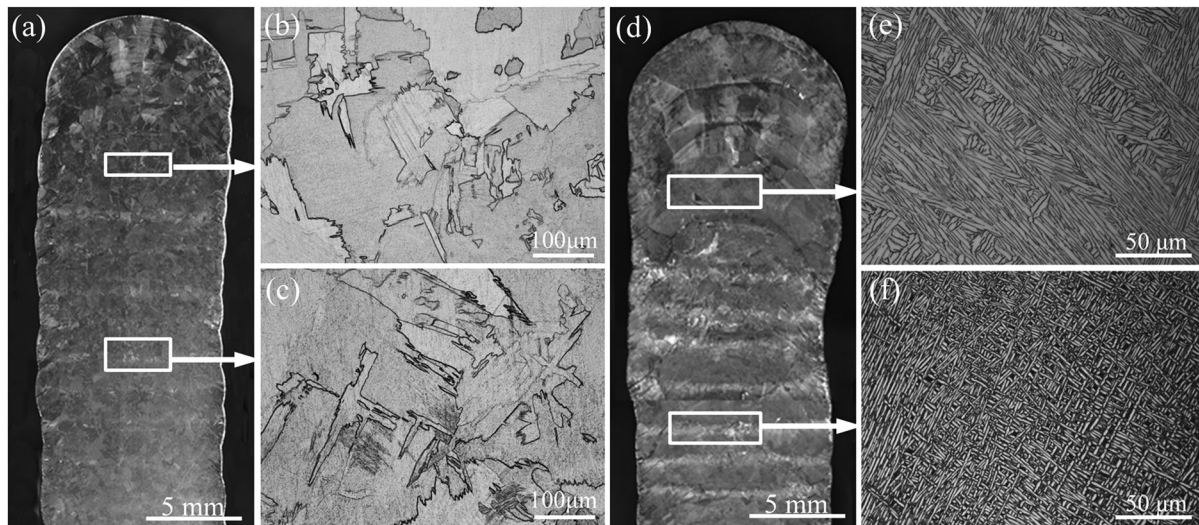


Figure 2. OM observation of cross-section microstructure of the deposited alloys: (a-c) Ti-6Al and (d-f) Ti-6Al-7Nb.

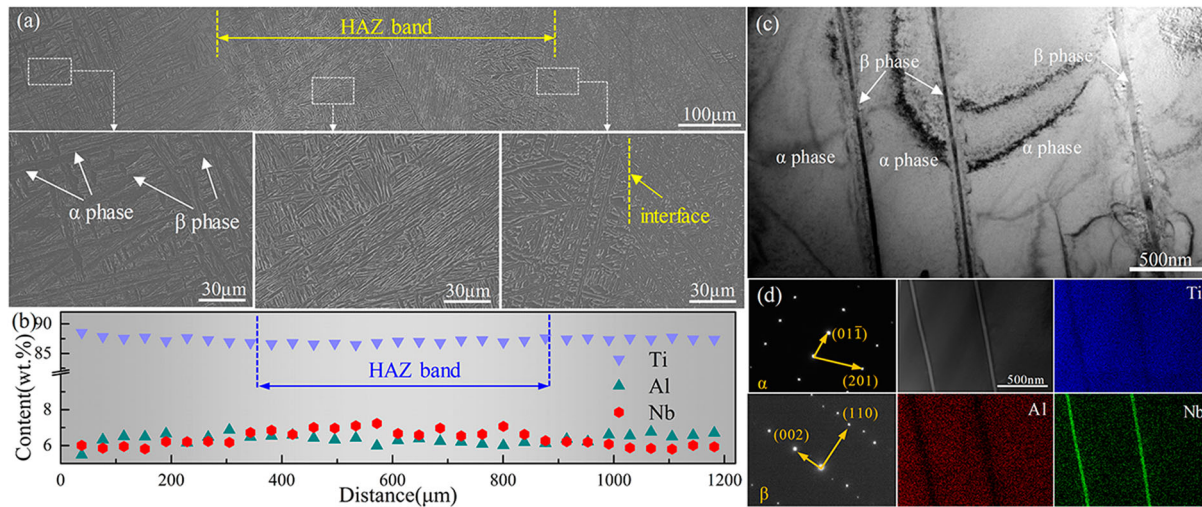


Figure 3. Characteristic of microstructure of Ti-6Al-7Nb alloy: (a) SEM observation of a complete HAZ band; (b) composition distribution across the HAZ band; (c) TEM observation of a random area in HAZ band; (d) corresponding EDS compositional maps and SAED patterns.

element. Figure 3(c) shows the TEM observation of β phase formed in the HAZ band, and the formation of nano-scaled lath β phase with a high concentration of Nb was proved by Figure 3(d). The microstructural evolution of the HAZ band could be attributed to the thermal cycle associated with the layer-by-layer deposition mode in WAAM, which will be further discussed.

Figure 4(a) is a schematic diagram of the temperature field in a given area of the deposited Ti-6Al-7Nb alloy under multiple heat input. As illustrated in Figure 4(b1–b3), after the solidification process of the molten pool with a composition of Ti-6Al-7Nb, solid-state phase transition of $\beta \rightarrow \alpha$ phase occurred during cooling stage. Meanwhile, Nb atoms in the α phase diffused towards β phase due to the low solid solubility of the Nb in the α phase, leading to the appearance of

a Nb-rich area near the phase interface. Moreover, the Nb concentration at the phase interface was gradually increased with the growth of α grains, which is conducive to the retention of β because Nb is a strong β phase stabilizing element. Thus, lath-like β phase remained between the α grains.

In the subsequent re-heating process, the phase transition of the deposited alloy could be divided into the following two cases. One occurred in a region where the reheating temperature was higher than the β phase transition temperature but lower than T_L . Take the region marked by red rectangular dotted frame in Figure 4(b3) as an example, the alloy here was fully transformed into β , as depicted in Figure 4(c1). Subsequently, similar phase transition of $\beta \rightarrow \alpha$ took place during cooling from the peak temperature. It is reported that the cooling rate,

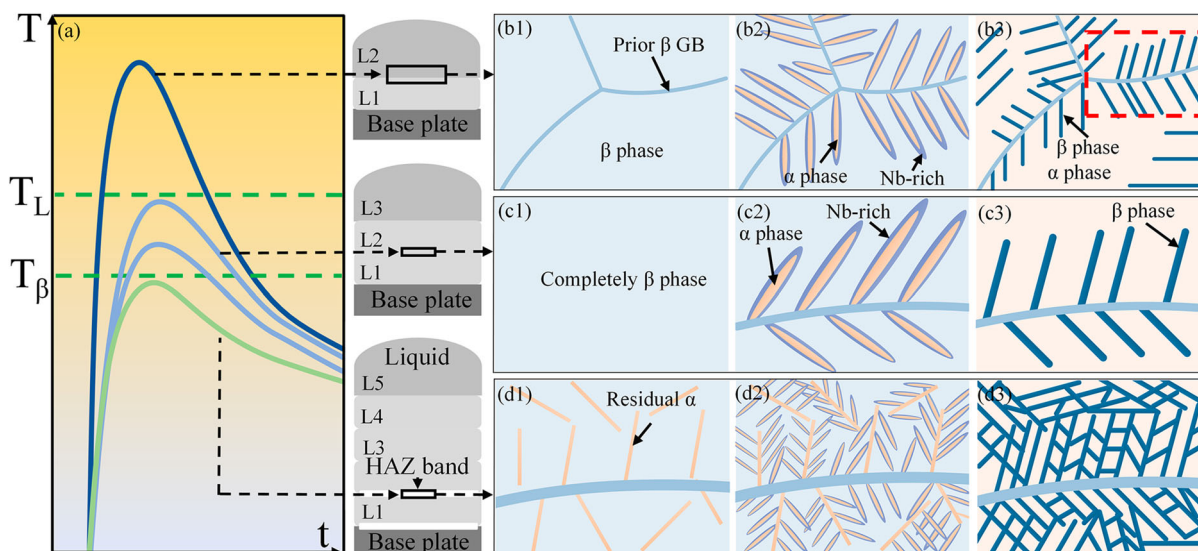


Figure 4. Schematic diagrams illustrating the possible phase transition of Ti-6Al-7Nb alloy: (a) thermal cycle curves corresponding to 4 times heat input; (b1–b3) phase transition of the alloy cooling from liquid phase; (c1–c3) phase transition of the region where re-heating temperature was over β phase transition temperature and (d1–d3) phase transition of the region where re-heating temperature was close to β phase transition temperature.

which is closely related to the alloy microstructure, did not change substantially for different thermal cycles during WAAM [22]. In this work, the interlayer temperature was fixed at 80°C. So, the transformed microstructure was similar to that cooling from molten pool since the same cooling rate, as shown in Figure 4(c2–c3).

The other occurred in a region where the reheating temperature was slightly lower than the β phase transition temperature. As the deposition height increased, the peak temperature of the alloy at the bottom was gradually decreased, leading to the existence of a banded zone with the reheating temperature slightly lower than β phase transition temperature. As described in Figure 4(d1), when the peak temperature approached to the β phase transition temperature, a small amount of α phase still remained in this zone. Once cooling took place, the secondary α phase began to precipitate and tended to grow along the pre- α phase as well as the grain boundaries of original β phase (Figure 4(d2)). Because the amount of secondary α phase formed was much higher than that of primary α phase, the microstructure was quite finer, as seen in Figure 4(d3). This banded zone is actually the HAZ band we discussed above. Based on this, it can be explained that HAZ band did not appear in the last four deposition layers was ascribed to fully β transition. In addition, the growth of β phase was not significant because the next peak temperature of the already formed HAZ band was low. Meanwhile, the diffusion of Nb atoms in the alloy was slow as a result of the low diffusion rate under a low temperature. Hence, the Nb was still concentrated in the original β phase region.

In conclusion, the biomedical Ti-6Al-7Nb alloy was successfully fabricated by a novel method of T-WAAM together with in-situ alloying. The deposited Ti-6Al-7Nb alloy showed Widmannstatten structure with the basket weave characteristics and periodic HAZ bands with a width of 400–500 μm appeared in the alloy except for the last four deposition layers. The microstructure with nano-scaled Nb-rich β phase in the HAZ band was finer than that in non-HAZ band. The HAZ band was a region where the reheating temperature was just slightly lower than the β phase transition temperature, in which a lot of secondary α phase along with the pre- α phase and original β phase formed. Compared to Ti-6Al alloy, the average ultimate tensile strength and elongation of Ti-6Al-7Nb alloy increased by 35% and 47%, respectively. The improvement of mechanical properties was ascribed to the grain refinement and the formation of nano-scaled lath β phase caused by the addition of Nb. This work provides a promising method to manufacture multi-element alloys (feasibility of Ti-48Al-2Cr-2Nb and Ti-6Al-4Nb was studied in Figure S4 and Figure S5), which is beneficial to expanding the application fields of WAAM.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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