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Hydrogen induced microstructure evolution of titanium matrix composites

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ARTICLE INFO

Article history:

Received 18 June 2017
Received in revised form
2 April 2018
Accepted 5 April 2018
Available online xxx

Keywords:

Titanium matrix composites
Microstructure evolution
Hydrogenation
Interface
Dynamic recrystallization

ABSTRACT

The effect of melt hydrogenation on microstructure evolution of Ti-6Al-4V matrix composites was investigated in this study. Molten alloy was hydrogenated with a mixture of hydrogen and argon, and reinforced at 5% total volume fraction with a 1:1 mol ratio mixture of TiB and TiC particles. Microstructure of as cast composites showed hydrogen induced more TiB whiskers with higher length-diameter ratio (LDR), because hydrogen accelerated atomic diffusion and then increased growth rate of TiB whiskers. Hot compression results indicated hydrogen reduced peak flowing stress. Microstructure of as compressed composites indicated hydrogen encouraged decomposition of residual lamellas. Hydrogen eliminated most cracks and holes along the interface between ceramic particles and matrix. Compared with unhydrogenated composites, the original ceramic particles in hydrogenated composites were fragmented into smaller pieces after compression. Electron back-scattered diffraction and transmission electron microscopy results indicated hydrogen increased volume fraction of dynamic recrystallization (DRX). And hydrogen decreased the density of dislocations nearby the interface.

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Introduction

Titanium matrix composites (TMCs) with high strength, high specific strength, high oxidation and corrosion resistance [1–4], have been considered as potential materials for high temperature application. *In situ* synthesized composites reinforced with ceramic particles have low cost and good interfacial bond, which have attracted much attention in

recent years. Among these composites, Ti-6Al-4V matrix composites reinforced with TiC and TiB particles have been proved to have excellent comprehensive properties [5]. However, the as cast composites can't be used directly due to poor ductility (less than 2%) [2]. Therefore, TMCs must be hot worked before use.

Compared with titanium alloys, TMCs have higher deformation resistance. Hydrogenation is an effective method to

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<https://doi.org/10.1016/j.ijhydene.2018.04.030>

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reduce deforming stress and improve hot workability. Wen [6] investigated effect of hydrogen on phase transformation and mechanical properties of TiAl based alloys, and found flowing stress of hydrogenated alloys was lower than that of unhydrogenated alloys, hydrogen impeded phase transformation from B_2 to w_0 by impeding the decrease of clearance radius of tetrahedron. Lu [7–10] hydrogenated TMCs reinforced with TiB and TiC particles, and found hydrogen decreased peak stress and increased super plasticity, and hydrogen also improved coordination of movement between matrix and ceramic particles during hot deformation. Usually, traditional hydrogenation is holding materials in hydrogen at high temperature (750 °C) for several hours (2 h), which means the alloys are in solid state [11]. This hydrogenation method is with low efficiency and longtime consumption, which can only be used to hydrogenate materials with thin thickness. Another hydrogenation method is called melt hydrogenation [12] which means melting alloys in gaseous mixture of hydrogen and argon, so synthesis and hydrogenation process can be completed at the same time. Wang [13] studied effect of melt hydrogenation on microstructure and hot deformation of Ti-6Al-4V alloy, and found hydrogen refined microstructure of as cast alloy and reduced flowing stress during hot deformation. Liu [14–16] calculated solubility of hydrogen in TiAl melt and studied the hot compression behavior, and found hydrogen encouraged decomposition of residual lamellas and increased volume fraction of dynamic recrystallization (DRX). Dong [17–19] studied effect of melt hydrogenation on glass-forming ability and mechanical properties of amorphous alloys.

In this study, hot workability of TMCs is decided by microstructure of matrix and distribution of ceramic particles together. Different with traditional hydrogenation, materials hydrogenated by melt hydrogenation are in liquid state [20], which means hydrogen has effect on distribution and morphology of ceramic particles. In our previous work [21], we have reported the hydrogen induced change on distribution of ceramic particles, this study focuses on effect of hydrogen on morphology of ceramic particles, microstructure of matrix alloy and their interface.

Materials and methods

Ti-6Al-4V matrix composites were reinforced at 5% total volume fraction with a 1:1 mol ratio mixture of TiB and TiC particles. The composites were *in situ* synthesized and hydrogenated by melting the raw materials in gaseous mixture of hydrogen and argon. Hydrogen percentage in gaseous mixture was 0, 10, 20 and 30%, respectively. In our previous work, we have reported the experimental details on control of hydrogen percentage in gaseous mixture and measurement of hydrogen content in composites [22], so this paper will not show further details. When hydrogen percentage in gaseous mixture was 10, 20 and 30%, hydrogen content in hydrogenated composites was 3.35×10^{-2} wt%, 5.31×10^{-2} wt% and 6.39×10^{-2} wt%, respectively.

The samples for microstructure observation of as cast composites were cut from center of as received materials. After mechanical grind and polish, the samples were etched

by solution of HF, HNO₃ and H₂O (volume fraction ratio was 1:1:8) for 10 s. Quanta 200F scanning electron microscope (SEM, secondary electron mode) was used to observe microstructure of as cast composites. Samples for hot deformation were cylinders with 6 mm in diameter and 9 mm in height, which were also cut from center of as cast composites. Hot compression was conducted on Gleeble-1500D machine, and compressing temperature was 850 °C and strain rate was 0.1, 0.01 and 0.001 s⁻¹, respectively. After compression, samples were quenched into water immediately to preserve high temperature microstructure. Microstructure observation of as compressed composites was conducted on Quanta 200F scanning electron microscope and FEI Talos F200X transmission electron microscope (TEM). Samples for TEM and electron back-scatter diffraction (EBSD) were cut from as compressed composites, and electrochemical polished in solution of 6% per-chloric acid, 34% *n*-butyl alcohol and 60% methanol (volume fraction) at -25 °C. Energy dispersion spectrometry (EDS) was performed on captured high angle annular dark field (HAADF) image under Scanning Transmission Electron Microscopy (STEM) mode. EDS and selected area electron diffraction (SAED) pattern were used to identify the ceramic phase in composites. And EBSD observation was conducted on Quanta 200F.

Results and discussion

Fig. 1 shows distribution and morphology of ceramic particles in as cast composites with increasing hydrogen content. Bright particles are reinforcements which distribute on gray Ti-6Al-4V matrix. Without hydrogenation, TMCs have homogeneously distributed ceramic particles, and there are no TiB whiskers with high length-diameter ratio (LDR) and no obvious prior β grain boundaries. In our previous work [22], we have reported that prior β grain boundaries became more obvious with increasing hydrogen content. This kind of microstructure change results from hydrogen induced superheat on melt surface. Higher superheat accelerates atomic diffusion and decreases nucleation rate of prior β phase. As results, the ceramic particles in hydrogenated composites gather around to form near network structure.

Similar microstructure can be observed in this study, especially in Fig. 1d, and the LDR of TiB whiskers increases with increasing hydrogen content. In this study, TiC has equiaxed form and TiB has needle-like shape. Their difference on morphology is decided by their crystal structure. TiC has no preferred growing direction and equiaxed growth rate, so it's easy for TiC to form as equiaxed particle. TiB has B27 crystal structure, and its fundamental unit cells are stacking of trigonal prisms with B atoms at center and Ti atoms at corners [23], and the unit cells are formed by stacking of trigonal prisms connected by their edges. This kind of stacking leads to zig-zag chain of B atoms along [010] direction, which decides TiB with whisker shape.

The LDR of TiB in hydrogenated composites is higher than that in unhydrogenated composites, which is more obvious in composites with 6.39×10^{-2} wt% hydrogen (shown as Fig. 1d). As discussed earlier, hydrogen improves superheat on melt surface, which increases growth rate of TiB, so the TiB in

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