Science Press

Trans. Nonferrous Met. Soc. China 17(2007) s471−s476

Transactions of Nonferrous Metals Society of China

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Effects of thermohydrogen treatment on microstructures of α+β and β titanium alloy as-cast

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Received 15 July 2007; accepted 10 September 2007

Abstract: The effects of thermohydrogen treatment on the microstructures of TC21 and Ti40 alloys as-cast were researched. The results show that the β phase content increases after charged hydrogen. Compound Ti_xH_y appears if H content reaches a certain content, which perfectly gathers on the grain-boundaries and/or dislocations and then diffuses into the grains. The microstructure of TC21 alloy after thermohydrogen treatment becomes fine and the best H content is 0.4% (mass fraction). However, the influence of thermohydrogen treatment on Ti40 microstructure is not obvious.

Key words: titanium alloy; thermohydrogen treatment; microstructure

1 Introduction

Ti and its alloys are widely used in many fields because of their excellent comprehensive properties. However, their low deformation limit, high deformation stress and easy cold fracture at room temperature (RT) restrict their processing technology at RT. Most of Ti-alloys must form at high temperature, but their deformation temperature is high, flow stress is large, and strain rate is low, especially for the Ti-alloys with high strength, high toughness, high modulus and heat resistance. In order to solve the problems of Ti-alloys during plastic deformation, two ways can be used. One is to increase the capacity of the instrument which is used and the other way is to decrease the deformation stress and deformation temperature. In 1959, Zwiecker and Schleicher, the former West Germany researchers, added suitable H content into Ti-alloy's ingot to research their hot processing properties. They got that the hot processing properties were improved greatly and then they proposed the idea of H increasing hot plasticity of Ti-alloys. After that, many research works on H changing hot processing properties of Ti-alloys were carried on[1−2]. KERR et al[3] suggested that the deformation stress of Ti-5Zr-9Al-5Sn-2Mo and Ti-9Al with H decreased greatly. The deformation stress of Ti-6Al-2Sn-4Zr-6Mo with 0.4% H at 730 ℃ was reduced by 30%−35%. In thermohydrogen treatment mainly H was used as a temporary alloying element. H can refine the microstructure[4−5] through H permeating, eutectoid decomposing, vacuum removing H. In this technology H was used to induce the plasticity and phase transformation, and H reversible reaction in Ti-alloys, which can get the optimum structure of Ti-H system improving the processing properties. SENKOV et al[6] and YOSHIMURA et al[7] presented a new system and way. Taking advantage of this technology they can receive the purposes of improving the processing properties, increasing the performance, decreasing the cost and promoting processing efficiency. In this article the H permeating behavior of Ti40 (a new full β Ti-alloy) and TC21 alloy (a new α+β Ti-alloy) as-cast at high temperature was researched and the effect of thermohydrogen treatment on their microstructures was analyzed. Both Ti40 and TC21 alloys were designed and developed in China. Ti40（Ti-25V-15Cr-0.2Si）is a new burn resistant titanium alloy, whose ingot is easy to fracture during hot deformation. TC21 is a new Ti-alloy with high strength, high toughness and damage tolerance. Both alloys have important application prospects. There are not any reports about the thermohydrogen treatment on these two alloys. Through researching their thermohydrogen treatment, some pieces of advice on the hot process of Ti40 and TC21 can be given.

Foundation item: Projects(50434030) supported by the National Natural Science Foundation of China

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2 Experimental

The materials used in this paper were commercial products of Ti40 and TC21 ingots. The H permeating technology was used for their casting samples. The dimensions of samples are as follows: 10mm in width, 10mm in length and 3 mm in thickness. H permeating temperature was below the phase transformation point. H content was from 0.1% to 0.5% (mass fraction). The vacuum removing H was carried out by annealing at 550 ℃ for 15 h (vacuum degree is 1.4×10^{-3} Pa).The phase analysis was performed the PW1700 X-Ray diffraction: using Cu target, at scanning rate of 2(°)/min. The optical microstructure (OM), scanning electronic microscope (SEM) micrographs and transmission electronic microscope (TEM) microstructure were separately carried out on OLMPUS PMG, S-2700 scanning electronic microscope and JEM-200CX transmission electron microscope. The etchant consists of 10%HF (volume fraction), 30%HNO_3 and 50%H_2O.

3 Results and discussion

3.1 Effect of H permeating treatment on microstructures of TC21 alloy

The casting microstructure of TC21 alloy is Widmannstaetten structure (Fig.1). It is coarse plate structure, whose characteristic is original β grains and there are clear α phases on the original β grain boundaries and plate α zone within the original β grain boundary. β phase is between α plates. The grain boundary α is wide, α zone is coarse and plate α is parallel(Fig.1(b)).

Figs.2, 3 and 4 show the microstructures of TC21 after charged hydrogen. It can be seen that the microstructures change greatly. α/β beam concentration is fine and close, and it is irregular. α plate is thin and α zone is small, which is owing to that H is a β stabilizing alloying element. With the increasing of H content, the content of β phase increases and the content of α phase is decreasing. The microstructural type is still Widmannstaetten structure, which mainly consists of α and β phases, and also little hydride α'' emerges (Fig.5). α'' phase nucleates and grows between α and β phases. If the H content is less, the grain boundary α changes little, as shown in Fig.4(a). With the increase of H content, α'' phase becomes coarse and α'' phase also appear at grain boundaries, leading to grain boundaries to be unclear, as shown in Fig.4(b). It is well known that the new phase appearance is decided by two factors.

Grain boundary energy and strain energy tend to be minimum, and so does α'' phase. If the H content is low, strain energy is the main factor. In order to reduce strain energy, α'' phase is in thin sheet structure between α and β phases. With the increase of H content, the content of

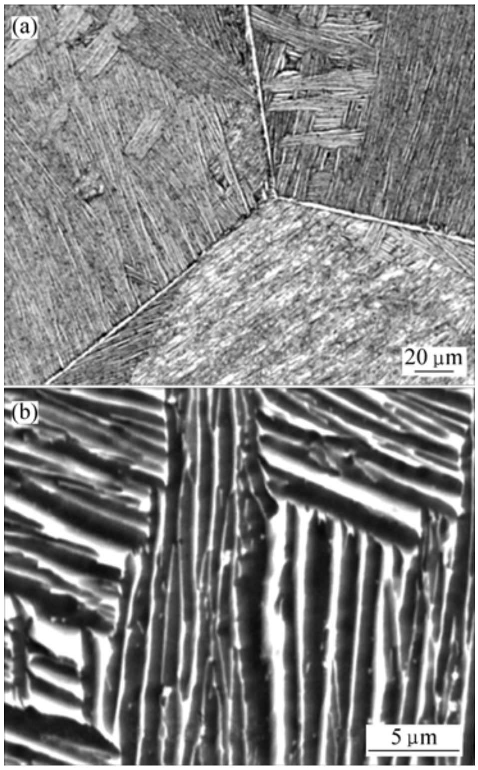

Fig.1 OM(a) and SEM (b) images of as-cast TC21 alloy

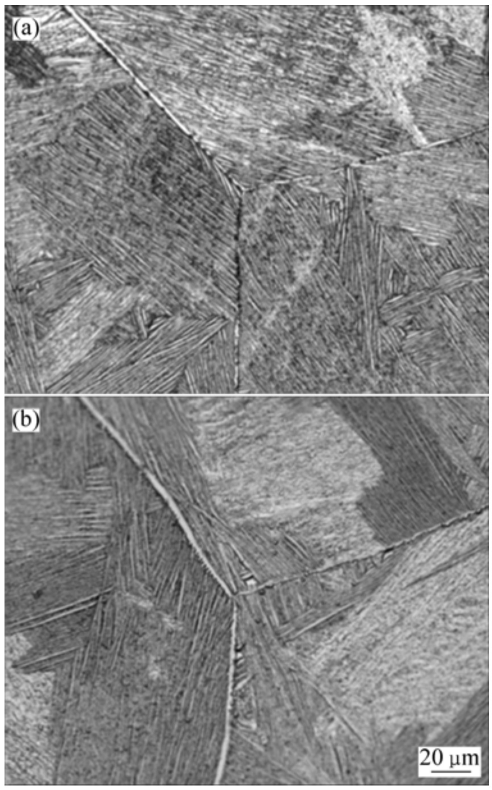

Fig.2 OM images of as-cast TC21 alloy after charged hydrogen: (a) 0.1%H; (b) 0.3%H

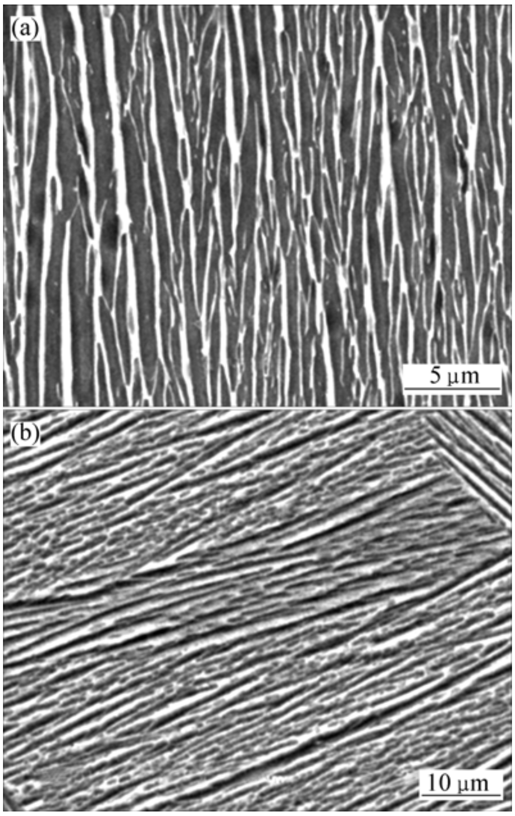

Fig.3 SEM images of as-cast TC21 after charged different contents of hydrogen: (a) 0.1%H; (b) 0.3%H

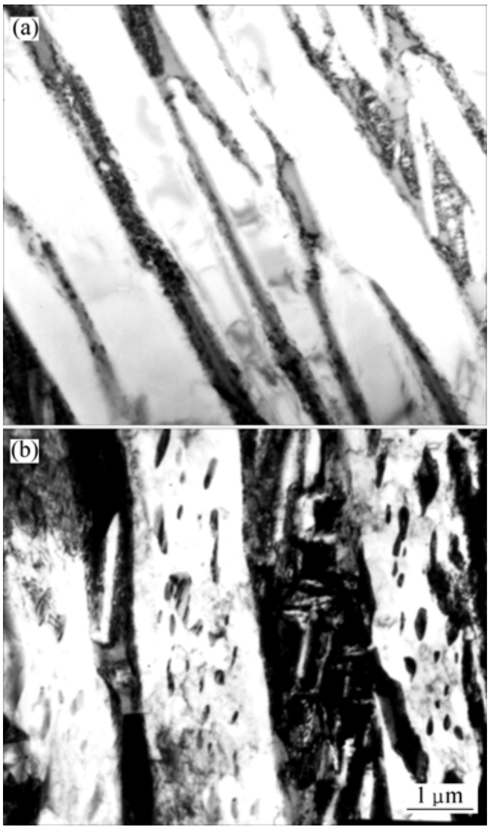

Fig.4 TEM images of as-cast TC21 after charged different contents of hydrogen: (a) 0.1%H; (b) 0.5%H

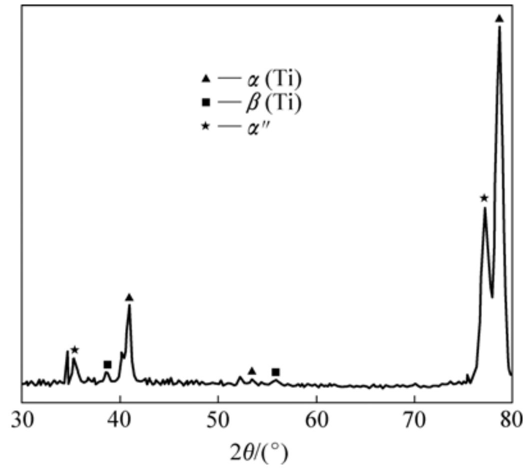

Fig.5 XRD patterns of as-cast TC21 after charged 0.5% hydrogen

α'' phase increases and grain boundary energy is the main factor. In order to decrease the surface energy, hydride becomes spherical, resulting in the minimum of the system free energy.

With the increase of H content, there are many dislocations and black points on the plate α phases (the white color in Fig.4) as shown in Fig.4. This is due to that the increasing β stabilization element H, β phases appear within plate α phase(the black point), causing the plate structure to be broken.

Figs.6 and 7 show the microstructures of as-cast TC21 after desorbing hydrogen. α/β plate mass zone within the original structure is broken and fine. The higher the H content, the clearer the breaking effect. H content of 0.4% has the best effect on the refining the microstructure and the original grain boundary becomes unclear (Fig.7(d)).

It is clear from Fig.7(c) and Fig.7(e) that α plate after desorbing hydrogen becomes fine and thin and it is also intermittent. Both α and β phases distribute evenly within the grains. The grain boundary does not change obviously, and the original long plate mass zone is still clear, which indicates that the fine grains after H treatment is limited in the plate α/β mass zone within the grains under these experimental conditions.

The results of XRD show that the casting structure of TC21 alloy consists of $\alpha+\beta$ one. After H treatment, the content of β phase increases and α phase decreases. And also there are some α''(TiH2) (Fig.5). After H treatment, lattices of α phase and β phase expand by 0.9% and 0.5%, respectively. H is a β stabilization element and it can reduce the phase transformation temperature of $(\alpha+\beta)/\beta$, resulting in increasing β phase content during annealing and quenching. Both HCP lattice of α phase and BCC lattice of β phase change greatly after H treatment. The peak of XRD moves to low angle, showing that the

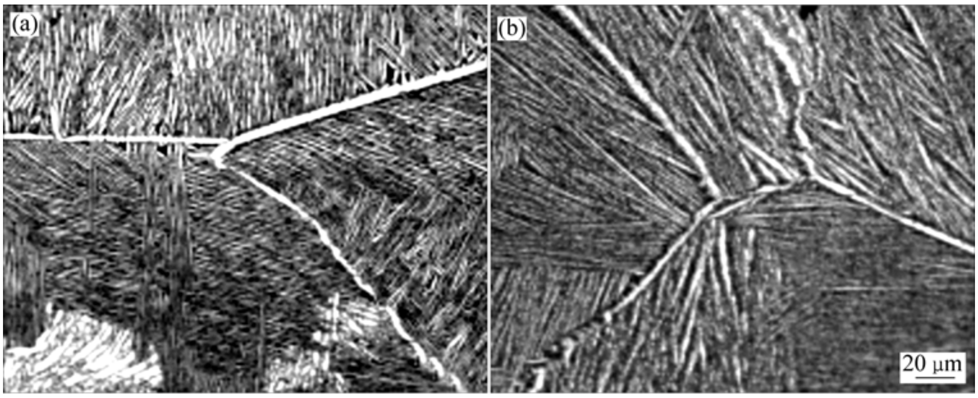

Fig.6 OM images of TC21 as-cast after desorbing hydrogen: (a) 0.1%H; (b) 0.4%H

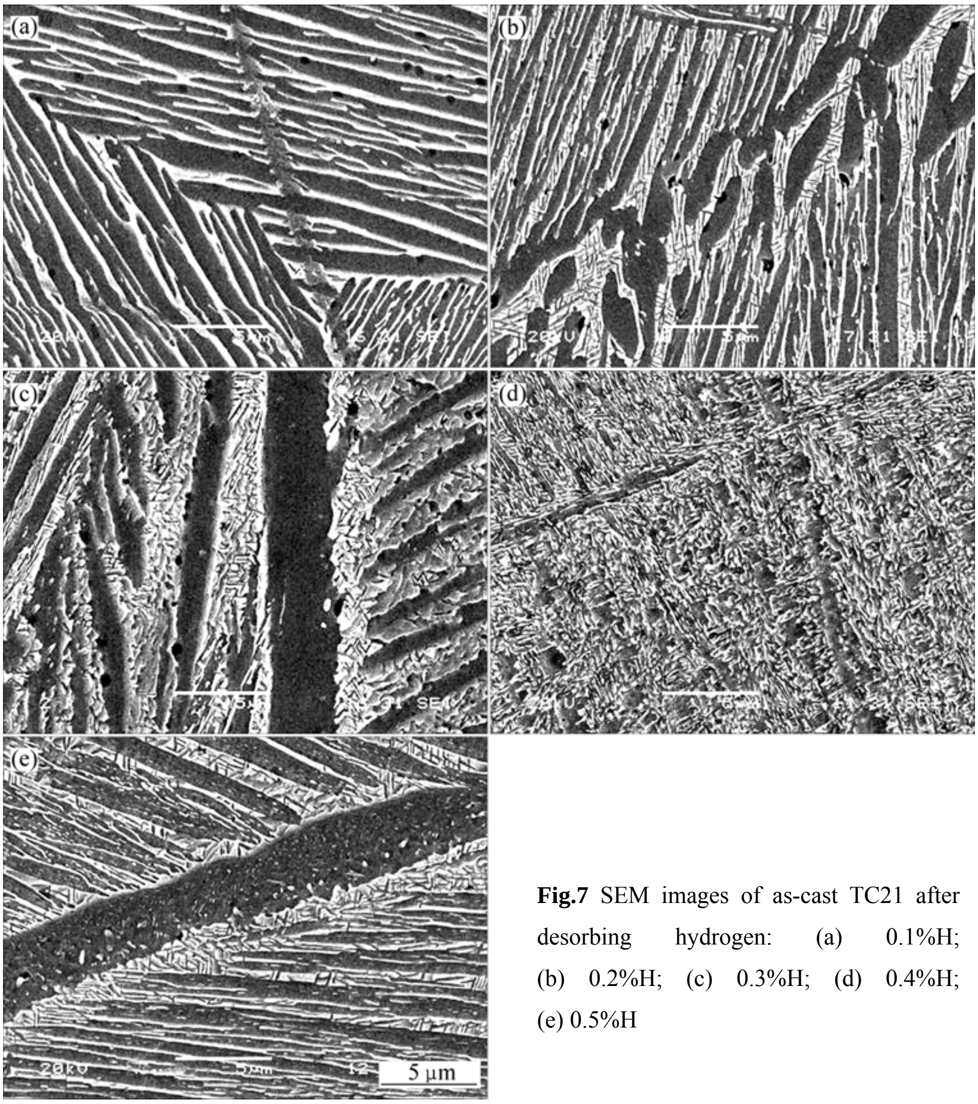

Fig.7 SEM images of as-cast TC21 after desorbing hydrogen: (a) 0.1%H; (b) 0.2%H; (c) 0.3%H; (d) 0.4%H; (e) 0.5%H

lattices of α and β phases expand. For the α+β Ti-alloys after H treatment, H mainly dissolves into β phase, leading to β phase lattice constant and specific volume to increase. This indicates that the changing rate of specific volume from phase transformation increases by 2%−5%(it is only 0.17%−0.25% without H treatment.), resulting in the effect of circulator heat treatment increasing greatly. And also for the different Ti-alloys, the changing rate of specific volume from δ hydride forming from matrix is as high as 17%−25%[8], resulting in large strain field around hydride and lots of dislocations within grains, which is similar to that of cold working[9]. H is removed and hydride decomposes in high temperature vacuum, and lots of excess distortion energy in matrix changes into vacancy. The interaction between dislocation and vacancy is good for the formation of subgrain and it becomes the recrystallization nucleus. While desorbing H the matrix emerges extensive recrystallization, leading to fine and equiaxed grains (Fig.7).

3.2 Effect of H permeating treatment on microstructures of Ti40 alloy

The casting microstructure of Ti40 alloy is single β structure (Fig.8). The microstructure of Ti40 after H treatment is shown in Fig.9. After H treatment, the bar-like precipitates appear and the amounts become more and more with increase of H content. The bar-like precipitates are TiH_2 after analysis. The hydride selectively gathers at the grain boundary and hydride will precipitate at the grain boundary when the H content is high enough, leading to coarse grain boundary. With the diffusion of H, also plate-like hydrides appear within the grains.

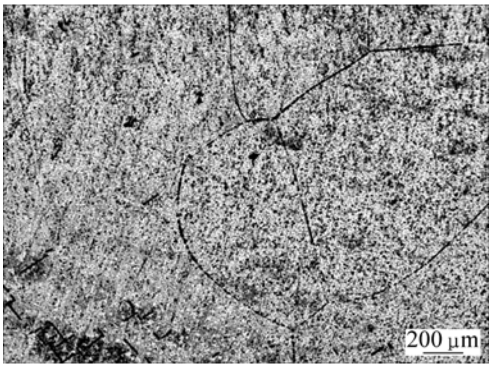

Fig.8 OM image of as-cast Ti40 alloy

After H permeating treatment, grain boundary becomes coarse and bar-like or sphere-like precipitates appear within grains (Fig.10). The analysis on the phases of different places in grain and at grain boundary by electron-probe shows that there is large difference of alloying element distribution among grain, grain boundary and precipitate. The content of β stabilization element at grain boundary and in precipitate is higher, which is due to the new distribution of main alloying elements in α and β phases from the H permeation. The H treatment is finished at 650 ℃. During this process, fine α precipitates will emerge.

The microstructure after desorbing H is shown in Fig.11. The original grain size does not change. The reason is as follows: Ti40 is a full β Ti-alloy. During H treatment, no phase changes besides little hydride in the structure. With the increase of H content, more and more precipitates at grain boundary emerge and grain boundary becomes coarse.

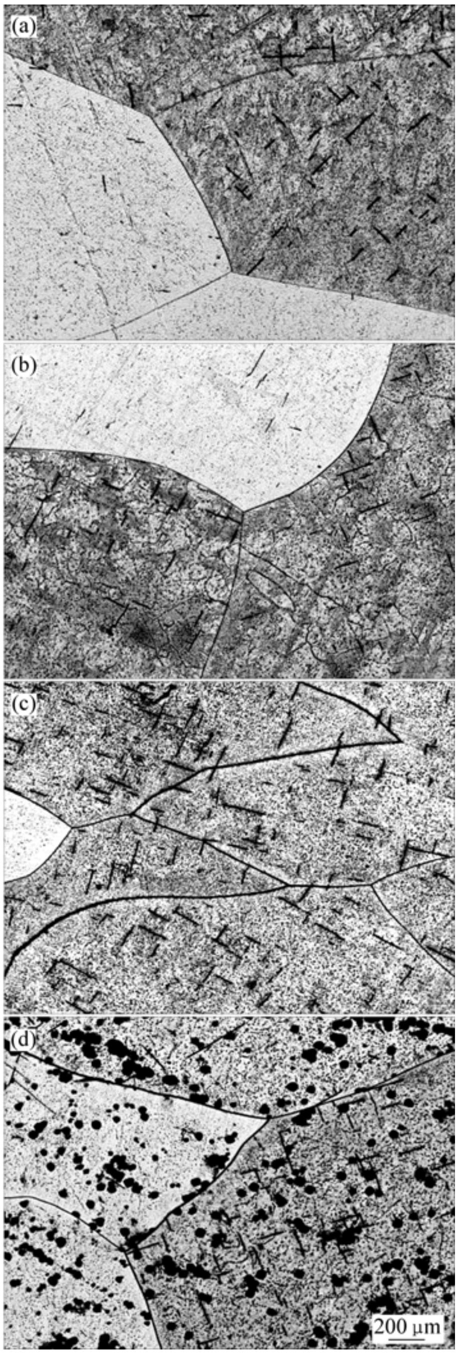

Fig.9 OM images of as-cast Ti40 after charged different contents of hydrogen: (a) 0.1%H; (b) 0.2%H; (c) 0.3%H; (d) 0.4%H

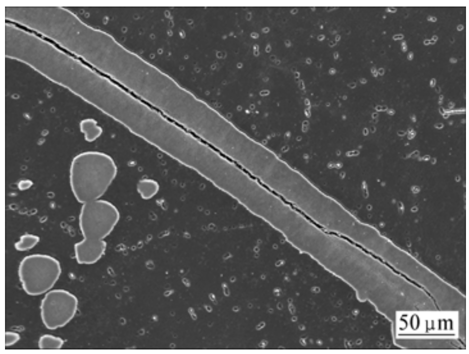

Fig.10 SEM image of as-cast Ti40 alloy after charged 0.5% hydrogen

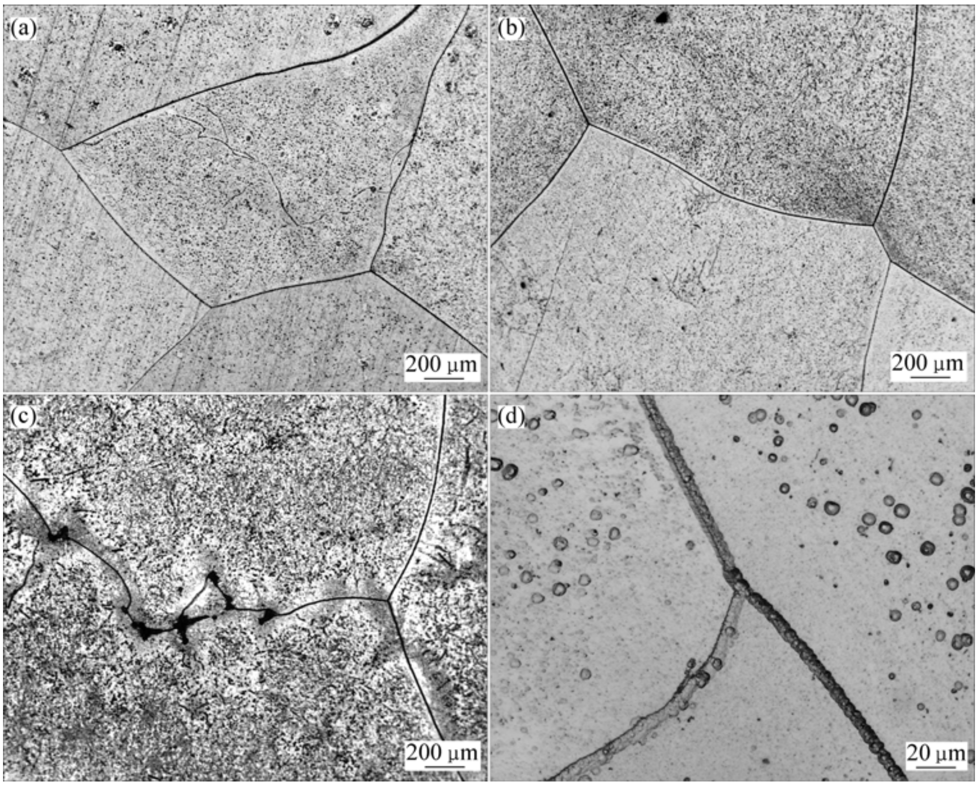

Fig.11 Optic micrographs of as-cast Ti40 after desorbing hydrogen: (a) 0.1%H; (b) 0.2%H; (c) 0.3%H; (d) 0.4%H

4 Conclusions

1) The content of β phase in TC21 and Ti40 alloys increases after H treatment. Hydride will emerge if H content is high enough and the hydride gathers selectively at grain boundary and/or dislocation.

2) The microstructure of TC21 is fined after H treatment. The best optimum fine effect is got when the H content is 0.4%. There is no obvious effect on Ti40 alloy.

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(Edited by LONG Huai-zhong)