

Effect of heat treatment on thermal stability of Ti40 alloy

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Received 15 July 2007; accepted 10 September 2007

Abstract: Five new heat treatment processes were designed, which were divided into three groups by their characteristics. The microstructures and mechanical properties of the alloy after the five heat treatments and thermal exposure at 500, 550 °C for 100 h were tested. The results indicate that a little differences exist in the performance of mechanical properties at room-temperature after the five heat treatments, and the thermal stability is the key factor for determining heat treatment process. Among the three groups of heat treatment processes, the best thermal stability is achieved after the first group of heat treatment. After annealing treatment at intermediate temperature, some defects and uneven grain boundaries are remained, which leads to the reduction fractions of precipitations on unit grain boundary and the harmful effect of precipitations on grain boundary is weakened. The process of annealing at 650 °C for 4 h is recommended the best heat treatment process for Ti40 alloy.

Key words: Ti40 burn resistant titanium alloy; heat treatment; thermal stability; mechanical property

1 Introduction

In China, Ti40 alloys have been researched in Northwest Institute for Nonferrous Metal Research(NIN) for many years and many achievements have been gained. By now, burn resistant mechanism[1–2], elevated temperature deformation mechanism[3] and mechanical properties and microstructures under different conditions[4–6] have been systemically researched. The further use of burn resistant titanium alloy in aero-engines requires that the burn resistant alloy can also be used at high temperature. And thermal stability is one of the key subjects for burn resistant titanium[7–8]. It has been proved that Ti40 alloy can be used at 500 °C for a long time. However, when the alloy is used at higher temperature the thermal stability can not satisfy the demands[6,9], for example at 550 °C. During thermal exposure process the formation of continuous precipitated zones along the grain boundaries is the main reason of the decrease of mechanical properties. In Ref.[10–11] the authors suggested that the adjustment of present heat treatment of Ti40 alloy may be an effective method to increase thermal stability. For this reason, in this paper five representative heat

treatment processes were purposefully designed aiming at improving thermal stability of Ti40 alloy. The mechanical properties and microstructures of the alloy after heat treatment and thermal exposure were tested. The results achieved in this paper will offer some theoretical and practical instructions for Ti40 alloy or other β alloy used at higher temperature. At the same time, this paper is also a proving and further research by the authors[11].

2 Experimental

500 kg Ti40 ingot was melted by vacuum arc remelting (VAR) in NIN of China. The rings were forged by special process, which was mentioned elsewhere[12]. Its composition is listed in Table 1. Tensile and thermal exposure samples were cut from the rings, the size for tensile and heat treatment blank samples were 12 mm×12 mm×70 mm. Before thermal exposure, all specimens were treated by different heat treatment process, which are listed in Table 2. After that, all specimens were

Table 1 Alloying compositions(mass fraction, %)

V	Cr	Si	Fe	N	C	O	H	Ti
25.77	14.70	–	0.09	0.01	0.01	0.07	0.001	Bal.

Table 2 Mechanical properties of Ti40 alloy after different heat treatments and thermal exposure

Group	Sample No.	Heat treatment	0 h				500 °C, 100 h				550 °C, 100 h			
			UTS/ MPa	YS/ MPa	El/ %	RA/ %	UTS/ MPa	YS/ MPa	El/ %	RA/ %	UTS/ MPa	YS/ MPa	El/ %	RA/ %
0	0 [#]	850 °C, 1 h, WQ+ 550 °C, 6 h, AC	970	940	23.5	42.0	1 053	1 025	5.5	9.0	755	–	1.5	2.0
I	1 [#]	600 °C, 4 h, AC	970	950	19.0	35.0	995	960	9.5	16.5	990	960	4.0	8.5
	2 [#]	700 °C, 4 h, AC	985	960	20.0	43.5	955	925	12.0	25.5	945	930	3.6	7.0
II	3 [#]	850 °C, 1 h, WQ+400 °C, 10 h, AC+600 °C, 5 h, AC	980	950	15.3	33.0	980	965	3.5	4.0	900	–	–	–
	4 [#]	900 °C, 1 h, WQ+400 °C, 10 h, AC+600 °C, 5 h, AC	980	940	18.0	34.0	935	–	–	–	860	/	1.0	2.0
III	5 [#]	900 °C, 1 h, AC+700 °C, 6 h, AC+560 °C, 5 h, AC	990	970	20.0	31.0	980	965	3.0	4.0	950	940	1.0	1.5

UTS is ultimate tensile strength; YS is yield strength; El is elongation; RA is reduction of area; “–” means that performance cannot be tested for complete brittle fracture.

separately thermal exposed at 500 and 550 °C for 100 h, and then the specimens were machined into 5 mm diameter standard tensile specimens for mechanical experiment with a gauge length 25 mm. All tensile tests including heat treatment condition (0 h) and thermal exposure condition (TST) were carried out on an Instron testing machine with a strain rate 10^{-3} s^{-1} . The optical microstructure (OM), transmission electronic microscope (TEM) microstructure and scanning electronic microscope (SEM) micrographs were separately carried out on OLMPUS PMG, JEM-200CX transmission electron microscope and S-2700 scanning electronic microscope. The etchant consists of 10%HF(volume fraction), 30%HNO₃ and 50%H₂O.

The heat treatment and thermal exposure process are listed in Table 2. 0[#] heat treatment process (G0) is recommended for Ti40 alloy at present in use.

3 Results and discussion

3.1 Mechanical properties

The mechanical properties of the alloy after various heat treatments and thermal exposure processes are shown in Table 2, and the ones after 0[#] treatment are present service performance for Ti40 alloy. It can be found that after 0[#] heat treatment (850 °C, 1 h, WQ + 550 °C, 6 h, AC) the plasticity of the alloy exposed at 500 °C for 100 h decreases sharply, but they still keeps to a certain degree (El=5.5%; RA=9.0%), which can be accepted and satisfy the requirement. When the thermal exposure temperature increases to 550 °C, the mechanical properties seriously deteriorated and yield strength can not be tested for complete brittle fracture. Also, ultimate tensile the strength(UTS) sharply decrease for precipitates on grains which were discussed in Ref.[11].

In this experiment five new heat treatment processes

can be divided into three groups, the first group (GI) is annealing treatment at intermediate temperature air cooling which includes 1[#] and 2[#] process; the second group (G II) is annealing treatment at higher temperature water quenching and followed by pre-ageing and followed by ageing which includes 3[#] and 4[#] process; and the third (G III) is annealing treatment at high temperature air cooling and followed by annealing at intermediate temperature and followed by ageing which includes 5[#] process. As shown in Table 2, after various heat treatments the mechanical properties (TST 0 h in Table 2) for the alloy show little difference, which indicates the mechanical properties at room-temperature produce little effect on the choice of heat treatment process, and thermal stability at high temperature is the key factor for heat treatment process. Judging by thermal stability for the alloy exposed at 500 and 550 °C for 100 h as shown in Table 2, it can be educed that GI process is better than others. And compared 1[#] and 2[#] heat treatment in GI, 2[#] process (700 °C, 4 h, AC) is more suitable. After 2[#] heat treatment the mechanical properties for the alloy exposed at 500 °C for 100 h is still excellent compared with the others, and when the thermal exposure temperature increasing to 550 °C, the mechanical properties can still satisfy the need. On the contrary, after G II and G III heat treatments, the alloy exposed at 500 °C is almost brittle failure, and when temperature increasing to 550 °C, it is complete brittle failure and some performance even can not be tested.

3.2 Microstructures

3.2.1 Microstructures of alloy after pre-aging

Fig.1 shows the OM and TEM images for the alloy after solution treatment at 900 °C for 1 h WQ followed by pre-ageing at 400 °C for 10 h AC. The pre-ageing treatment (ageing at 400 °C for 10 h AC in 3[#] and 4[#] process) aims at adjusting the sites of precipitates and

optimizing thermal stability which was suggested in Ref.[11]. But the results (Fig.1) show that pre-ageing treatment produced little effect on microstructure, besides some dislocations there was no any transition phase no matter inside grains or on grain boundaries, which may be due to the structure stability of Ti40 (high Mo-equivalent leads to a high stability of β matrix).

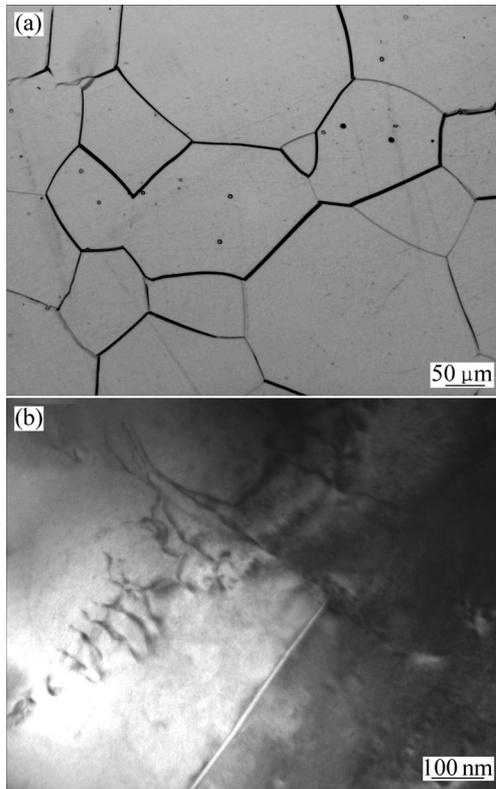


Fig.1 OM(a) and TEM(b) micrographs of alloy after pre-ageing at 900 °C for 1 h WQ followed at 400 °C for 10 h AC

3.2.2 Microstructures after heat treatment and thermal exposure

Fig.2 shows the OM microstructures of alloy after heat treatment (0[#], 1[#], 4[#] and 5[#] technology) and thermal exposure at 550 °C for 100 h (these microstructures are separately representation microstructures of G0, G I, G II and G III). Compared the microstructures among various treatments of 0[#], 4[#] and 5[#] process, it can be found that no matter after heat treatment or thermal exposure their microstructures were almost the same. Heat treatment produced the coarse equiaxed grains and there were little precipitates in the microstructures, the sequent thermal exposure process led to more precipitates in the microstructure, and which mainly distributed on grain boundaries. In Ref.[11] the authors discussed the amount and sites of precipitates in Ti40 alloy during thermal exposure process at 550 °C after 0[#] heat treatment, and pointed out that special composition (higher Mo-equivalent) and high thermal exposure temperature led to more precipitates on grain

boundaries and which weakened the strength of grain boundaries and deteriorated thermal stability. From the mechanical properties listed in Table 2 it can be seen that after various heat treatments besides 1[#] and 2[#] process the UTS decreased to a certain degree for the alloy exposed at 550 °C for 100 h, which also was resulted from the much more precipitates on grain boundaries. However when the alloy exposed at 500 °C for 100 h there was little change for UTS, which indicates that the precipitates on grain boundaries is not so much that decreasing UTS, they only produce obviously effect on plasticity (as shown in Table 2).

Compared with 0[#], 4[#] and 5[#], the microstructure of 1[#] specimen is obviously different (the microstructure of 2[#] specimen is similar to the ones of 1[#] specimen). It can be found that curve grain boundaries exist and much forging mark still keeps inside the grains. After thermal exposure the size of grains and morphology of grain boundaries keeps unchanged and more precipitates exist inside the grains and on the grain boundaries compared with the as-heat treated specimen (Fig.1(b)). By the mechanical properties in Table 2 it can be thought that the alloy achieves better thermal stability with this microstructure and the precipitates on the grain boundaries produce weaker effect on the mechanical properties. On the contrary, the microstructure with smooth grain boundaries after 0[#], 3[#], 4[#] and 5[#] heat treatment leads to bad thermal stability.

3.3 Fractographs of alloy

Fig.3 is SEM fractographs of the alloy after various heat treatment and heat exposure (1[#], 4[#] and 5[#]). It can be shown that there is little difference between the fractographs with the microstructure of smooth grain boundaries (the specimens of 4[#] and 5[#]). For the fractographs of as-heat treated specimens (Fig.2(c) and (e)), the cracks are originated from grain boundaries and the better plasticity is resulted from the dimples in transcrystalline fracture inside grains. After thermal exposure at 500 °C for 100 h (Fig.3(h)), the transcrystalline dimples rapidly decreased, they only could be found in few grains, which indicate that a number of precipitates have been formatted on grain and they weaken the grain boundaries strength and led to much more intergranular brittle fracture. When the thermal exposure temperature increasing to 550 °C (Fig.3(d) and (f)), the fractographs shown complete brittle fracture and grain edges could be clearly found. However, the fractographs show much difference of 1[#] specimen compared with the ones of 4[#] and 5[#]. After being thermal exposed at 500 °C for 100 h (Fig.3(g)), although the fractographs still show much intergranular fracture, many shallow dimples distribute on the fracture surface of intergranular section, this fractographs is

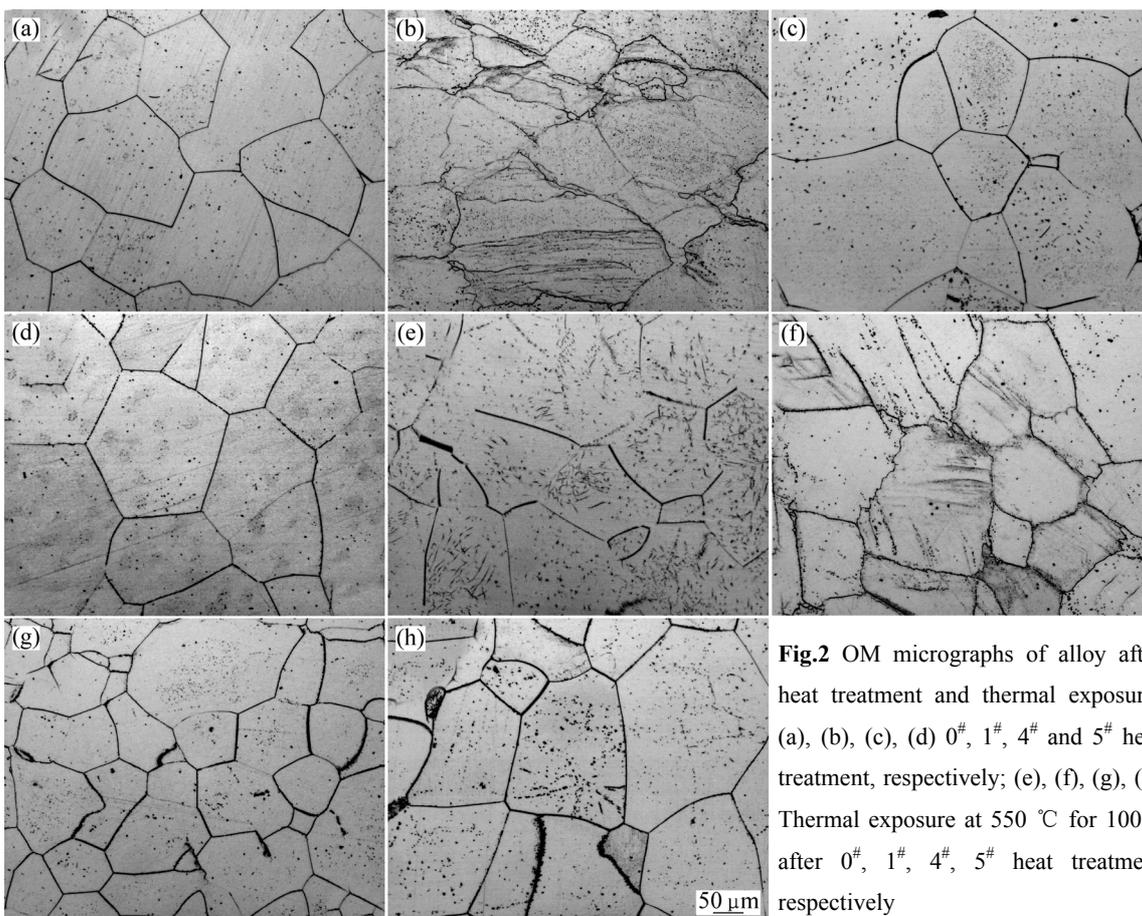


Fig.2 OM micrographs of alloy after heat treatment and thermal exposure: (a), (b), (c), (d) 0[#], 1[#], 4[#] and 5[#] heat treatment, respectively; (e), (f), (g), (h) Thermal exposure at 550 °C for 100 h after 0[#], 1[#], 4[#], 5[#] heat treatment respectively

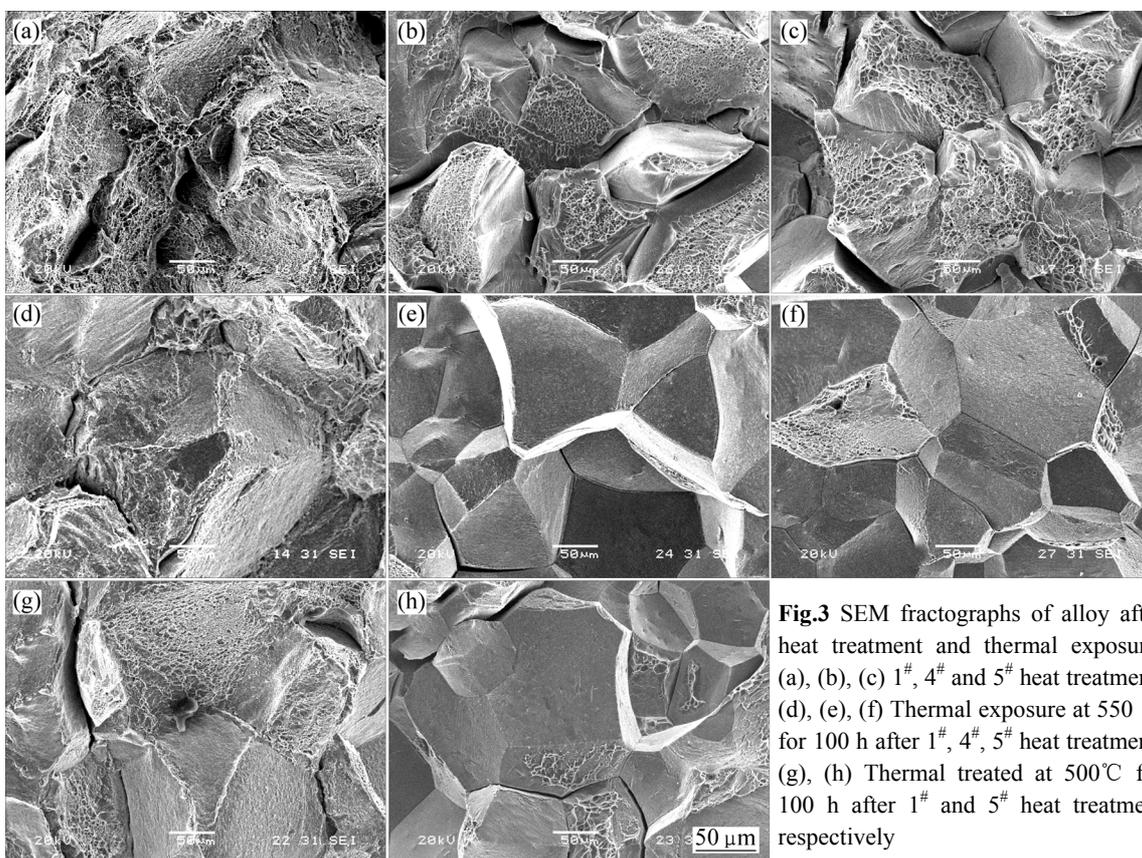


Fig.3 SEM fractographs of alloy after heat treatment and thermal exposure, (a), (b), (c) 1[#], 4[#] and 5[#] heat treatment; (d), (e), (f) Thermal exposure at 550 °C for 100 h after 1[#], 4[#], 5[#] heat treatment; (g), (h) Thermal treated at 500 °C for 100 h after 1[#] and 5[#] heat treatment respectively

corresponding with its mechanical in Table 2. When the thermal exposure temperature increasing to 550 °C, there were riverlet patterns on the fracture surface and tearing mark on grain edges unlike the ones of G II and G III process with bright sharp fracture surface, which indicates precipitates on grain boundaries during thermal exposure process produced weak effect on mechanical properties after G I treatment.

4 General discussions

4.1 Sites precipitation

The authors ever pointed out that it was an important characteristic that precipitates formed on grain boundaries during thermal exposure process, which was an important factor of deteriorative thermal stability[11]. It is suggested that the sites of precipitates could be adjusted by pre-ageing treatment. By pre-ageing treatment at low temperature some metastable phases (ω or β') would maybe dispersedly precipitate inside grains and the sequent thermal exposure at high temperature would lead to precipitates nucleating at the sites of metastable phases or the metastable phases directly transformed into α phase, a structure with homogeneous distribution of α phase inside grains could maybe achieve and it was prospective to improve the thermal stability. In this paper the pre-ageing treatment was applied in G II process (3[#] and 4[#] heat treatment). But the experimental results (Table 2 and Fig.1) show that the pre-ageing treatment at 400 °C for 10 h produces little effect on the microstructures and mechanical properties for Ti40 alloy, which may be useful to other near- β alloy. Judging by the mechanical properties and microstructures (Table 2 and Fig.2), it can be found that the key factor of determining heat treatment process is the morphology of grain boundaries in microstructures after heat treatment. Although the heat treatment of G0, G II and G III are different, their microstructures after heat treatment all show smooth grain boundaries, the effect of their microstructures on thermal stability is similar. However, after G I heat treatment (1[#] and 2[#]), because of lower annealing temperature, some forging marks were reserved and many working defects (for example dislocations, twin, etc) inside grains cannot be complete eliminated, they offer the effective nucleation sites for precipitates during thermal exposure processes, which decrease the tendency of precipitates nucleating on grain boundaries and optimize the distributions of precipitates during thermal exposure process leading to a better thermal stability.

4.2 Amount precipitation

The other way of decreasing the effect of precipitates on grain boundaries is to reduce the amount

of precipitates on unit grain boundary. After G I heat treatment, the microstructure shows curve unsmooth grain boundaries which can increase the effective area of grain boundaries and consequently the amount of precipitates on unit grain boundaries will reduce, which may be the main reason that the morphology of grain boundaries play the main role for thermal stability. Although the heat treatment processes of G0, G II and G III are different, their annealing temperature is higher and diffusion is sufficient which lead to sharp and smooth grain boundaries. During thermal exposure process, once precipitates nucleating on grain boundaries, they will rapidly growth along grain boundaries and finally form complete precipitates zone along grain boundaries, which is clearly shown in the fractographs of Fig.3(d), (f) and (h) and discussed in Ref.[11]. On the contrary, after G I heat treatment, fracture surfaces are rugosities and show visible tearing marks (Fig.3(b) and (g)), which indicates the amount of precipitates on unit grain boundaries is little compared with others treatment.

4.3 Best annealing temperature

Judged by the above analysis, it can be found that the annealing treatment at medium temperature is more acceptable, and there is a best annealing temperature for Ti40 alloy. If the temperature is lower than this temperature, the annealing process will cannot sufficiently adjust forging microstructures, which maybe lead to hidden trouble in application process. However, when the temperature is higher than this temperature, because of the high diffusion coefficient, the defects inside the grains will reduce and grain boundaries become smooth, which will increase the amount of precipitates on unit grain boundaries during thermal exposure process and leads to a bad thermal stability. Thus, judged by the mechanical properties and the above analysis, we think that the annealing treatment at 650 °C for 4 h air cooling is the best heat treatment for Ti40 alloy.

5 Conclusions

1) A little difference exists in the mechanical properties for the Ti40 alloy at room-temperature after various heat treatments, which produce little effect on the choice of heat treatment process.

2) For different kinds of heat treatments, the annealing at medium temperature is the best heat treatment for thermal stability of Ti40 alloy. After annealing at medium temperature, the alloy reserves some forging defect inside the grains and the microstructures show curve unsmoothed grain boundaries, which reduces the precipitates on unit grain boundaries during thermal exposure and lead to a weak

effect of precipitates on mechanical properties.

3) Judged by the mechanical properties and the microstructure of alloy, annealing at 650 °C for 4 h air cooling is the best heat treatment for Ti40 alloy.

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(Edited by YANG Hua)