TMS2014 Annual Meeting Supplemental Proceedings
TMS (The Minerals, Metals & Materials Society), 2014

STRUCTURE-PROPERTY CORRELATION IN AN AIRCRAFT SHEET METAL ALLOY Ti-15V-3Cr-3Al-3Sn

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Keywords: Beta titanium alloy, Duplex aging, Age hardening, Microstructure, micro Vickers hardness, Tensile testing, Fractography.

Abstract

This paper presents the age hardening behavior of solution treated metastable beta titanium alloy Ti-15V-3Cr-3Al-3Sn (Ti-15-3) as monitored by hardness measurements, tensile testing and microstructural examinations. The alloy was subjected to various single and duplex aging treatments. Single aging was carried out in the range from 200-550 °C for times up to 150 h. For the duplex aging first step aging was performed in two different ways. (i) 24 h at 250 °C (ii) 10 h at 300 °C ; second step aging was carried in the range 350-500 °C, for times up to 150 h. In general, duplex aging treatments resulted in higher hardness values compared to single step aging. Amongst all the duplex aging treatments, two aging treatments – 24 h / 250 °C followed by 8 h / 500 °C and 10 h / 300 °C followed by 10 h / 500 °C resulted in good balance of tensile strength and ductility; however, only the former treatment resulted in homogenous precipitation with no precipitate free zones.

1. Introduction

Metastable beta titanium alloys have been developed to overcome the shortcomings of alpha (α) and alpha+beta ($\alpha+\beta$) titanium alloys – low hardenability, inadequate deformability and poor machinability. The alloy Ti-15V-3Cr-3Al-3Sn (Ti-15-3) is one of the beta alloys being commercially produced and has been finding increasing application in aerospace due to its superior cold formability at room temperature, high strength after aging, deep hardenability, excellent corrosion resistance. The excellent cold formability enables production of this grade in the form of thin strips and sheets. The grade Ti-15-3 found many applications in aircraft and other industry sectors - airframe structure, ducts, fire extinguishers, body armour, springs, fasteners, foil, clips, brackets and metal matrix composites [1].

The nature of precipitation of α phase has a strong influence on the mechanical behavior of β titanium alloys [2-5]. Duplex aging is known to lead to improved performance of metastable β titanium alloys through homogeneous α precipitation. For example, studies by Krugmann and Gregory [2] and Wagner and Gregory [3] proved that duplex aging of the metastable β-Ti alloy Ti38-644 (β-C) leads to a more homogenous distribution of α precipitates. Schmidt et al [4] showed that carefully designed duplex aging treatments can improve the tensile ductility and high cycle fatigue life of β-C alloy. Furuhara et al [5] brought out that a low temperature – high temperature two step aging treatment of Ti15-3 alloy results in a more uniform and finer distribution of α precipitates than when single step aging is carried out. These authors have also shown that higher hardness levels can be obtained by resorting to two stage aging.

There is a need to identify optimum two step aging treatments for Ti-15-3 alloy, aiming at substantially superior combination of mechanical properties than what is possible with single step aging and a microstructure devoid of features which are known to adversely affect the fatigue life. The objective of the research reported here has been to establish an optimized two step aging cycle for the alloy Ti15-3 which yields a good strength-ductility combination and a microstructure conducive to a high fatigue limit. Detailed studies on mechanical properties obtained after single step aging and two step aging have been reported elsewhere [6]. The abbreviations SA and DA shall be used in this paper to denote single aging and two step / duplex aging respectively.

2. Material and Experimental Methods

The Ti15-3 alloy used in the present study was supplied in the form of rods 16 mm in diameter by GE Wick, China. The alloy was in the solution annealed condition; solution annealing was carried out at 850 °C. The detailed chemical composition is given in Table 1.

Table 1. Chemical composition of the alloy

V	Cr	Sn	Al	Fe	O	H	N	C	Ti
15.0	3.1	3.0	2.7	0.18	0.09	0.003	0.027	0.008	Bal

Discs of 6 mm in height were cut from the as-received rods using electro discharge wire cutting machine for both the SA and DA studies. Aging was carried out in an inert atmosphere furnace which is heated by SiC heating elements and the temperature was controlled within ± 2 °C. SA studies were carried out in the temperature range 200-550 °C, at 50 °C temperature interval. In the case of DA, two different cycles were used for the first step aging (i) 24 h at 250 °C (ii) 10 h at 300 °C, while the second step aging was carried out in the temperature range 350-500 °C at 50 °C temperature interval.

In both SA and DA, age hardening was monitored by carrying out micro Vickers hardness measurements, tensile testing and microstructural examinations. Hardness was measured using a standard Vickers diamond pyramid hardness tester with 500gm load and dwell time of 10 seconds on a metallographically polished specimens. The hardness values reported in the paper are average of at least 10 random measurements made on the sample.

Tensile testing in select cases was performed at room temperature to supplement the hardness tests to monitor the age hardening reaction taking place during SA and DA. Round tensile test specimens conforming to ASTM E8M requirements were used. Tensile testing was performed using electromechanical universal testing machine supplied by TE, China. Testing in any given condition was carried out on at least triplicate number of specimens and the test results reported herein are the average values.

Special etching was carried out using Ammonium bifluoride (NH_4HF_2) based etchant, prepared by dissolving 5 grams of NH_4HF_2 in 100 ml of distilled water for optical microscopic examination.

Metallographic polished specimens were etched with Kroll's etchant were used for microstructural examinations. Microstructural examination of select specimens in the aged condition was carried out using Zeiss-Supra™ 55 Field Emission Scanning Electron Microscopy (FESEM) with Back Scattered Electron Imaging (BSEI). Fractographic studies were carried out on broken tensile test samples using the FESEM in Secondary Electron Imaging (SEI) mode.

3. Results and Discussion

3.1 As-received Condition

The grains of the as-received condition (solution treated at 850 °C) rod were equiaxed with 35μ grain size, and the hardness of the material was measured to be 259 HV. The XRD pattern confirmed that the microstructure consists of single phase beta.

The starting microstructure, i.e., the microstructure after solution treatment and before any aging was done, has an important influence on the fatigue life of the alloy in the aged condition. In particular, unrecrystallized beta grains if any can lead to heterogeneous precipitation of alpha phase [7]. Studies on β-C alloy have revealed that a completely recrystallized microstructure results in better homogeneity of alpha phase precipitation and consequently a better fatigue limit [7].

The as-received material was subjected to decoration aging for 2 h at 482 °C followed by special etching and metallographic examination. Decoration aging followed by this etching using ammonium biflouride is very effective in revealing unrecrystallized regions, if any present, in the microstructure [7]. Such regions appear dark. Fig. 1 shows the microstructure of the Ti15-3 alloy. It could be clearly demonstrated that there were no unrecrystallized regions in the material in the as-received condition.

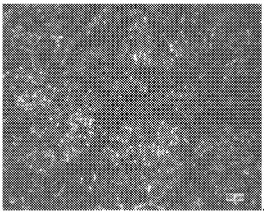


Figure 1. Optical micrograph after special etching of the as-received alloy

3.2 Single Aging Studies

Progress of decomposition reaction during single aging was monitored through micro Vickers hardness measurements. Aging temperatures were in the range 200-500 °C at 50 °C interval. The cumulative aging time was in the range 20-150 h depending on the aging temperature. For aging at 200 °C and 250 °C, there was no perceptible change in hardness even after 150 h of aging. Aging at 300 °C and 350 °C resulted in monotonic increase in hardness upto 150 h. At 400 °C and 450 °C, material is already in overaged condition after 50 h. At 500 °C, 20 h was sufficient to overage the material. Details of age hardening during SA have been presented elsewhere [6]. Details of tensile tests carried out as a function of aging temperature have been given elsewhere [6]. Aging at 300/350 °C leads to near-zero or nil ductility. Adequate ductility (% elongation ≥7 and % reduction in area ≥25) could not be obtained when aged at ≤450 °C. Aging at 500 °C resulted in good balance of strength and ductility.

3.3 Duplex aging with 24 h/250 °C Adopted as First Step

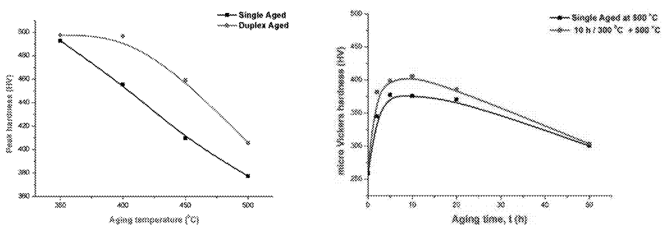
Limited experimentation was carried out with 24 h/250 °C +8 h/500 °C chosen as DA treatment and the results were compared with those obtained after 8 h/500 °C SA treatment. Results of tensile testing and hardness in the two conditions are compared in Table 2. The hardness, UTS and YS after SA are significantly higher in the DA condition; however, the elongation and reduction in area values are somewhat lower.

Table 2. Comparison of mechanical properties after SA and DA with 24 h / 250 °C as the first step for DA

Condition	Heat treatment details	micro Vickers hardness (HV)	UTS (MPa)	0.2% YS (MPa)	EL (%)	RA (%)
SA	8 h/500°C	358	1193	1120	10.0	35.5
DA	24 h/250°C + 8 h/500°C	376	1269	1201	8.0	28.5

3.4 Duplex Aging with 10 h/300 °C Adopted as First Step

Second step aging was carried out in the temperature range 350-500 °C at 50 °C interval. Hardness was found to monotonically increase up to a cumulative aging time of 150 h when aged at 350 °C. However, when aged at 400 °C and 450 °C, the material appears to have transformed into an overaged condition after 50 h of aging. Overaging sets in even earlier when aged at 500 °C; after 20 h of aging, the material is already in overaged condition. Peak hardness values obtained after DA were higher than the peak hardness values obtained after the counterpart SA. Fig. 2 (a) brings out the comparison. Fig. 2 (b) shows aging curves for SA at 500 °C and DA (10 h/300 °C +aging at 500 °C) superimposed. It can be seen that DA results in higher peak hardness, peak occurring in similar aging time for SA and DA.



Figures 2. (a) Peak hardness values obtained during SA and DA as a function of aging temperature (b) Hardness variation curves during SA and DA superimposed

Details of tensile testing carried out as a function of second step aging temperature are presented elsewhere [6]. Second step aging at 350 °C resulted in near-zero or nil ductility condition. Second step aging at ≤450 °C did not result in good balance of strength and ductility. Second step aging at 500 °C for 10 h, gave high strength with respectable ductility (8.6% elongation and 34.5%

reduction in area). Results of hardness and tensile testing in 10 h/500 °C SA condition and 10 h/300 °C +10 h/500 °C DA condition are presented in Table 3. Hardness after DA is higher by 32 HV. The UTS and YS values are also higher by 33 and 37 MPa respectively, with ductility also showing a little improvement.

Table 3. Comparison of mechanical properties after SA and DA with 10 h / 300 °C as the first step for DA

Condition	Heat treatment details	micro Vickers hardness (HV)	UTS (MPa)	0.2% YS (MPa)	EL (%)	RA (%)
SA	10 h/500°C	373	1193	1131	7.5	31.0
DA	10 h/300°C + 10 h/500°C	405	1226	1168	8.6	34.5

3.6 Microstructural Analysis using FESEM

Microstructural examination was carried out using FESEM on samples which received the treatments listed in Tables 2 and 3. Two samples were in SA condition, while the other two were in DA condition. One SA sample was aged at 500 °C for 8 h, while the other was aged at the same temperature for 10 h. One DA sample received the treatment 24 h at 250 °C + 8 h at 500 °C, while the other was subjected to 10 h at 300 °C + 10 h at 500 °C. The two DA treatments were found to give optimum balance of strength and ductility as brought out in the foregoing sections. Figs. 3(a) to 3(d) give representative SEM photographs corresponding to these four conditions. In all cases one can observe a high volume fraction of α phase that has precipitated out of β phase. Side plates of α phase were seen starting from some of the β grain boundaries and spanning into the grain interior in the two DA conditions and 10 h/500 °C SA condition. Fig. 3(a) shows an example of such plates observed in SA 10 h/500 ° C conditions.

3.6.1 Precipitate Free Zones (PFZs)

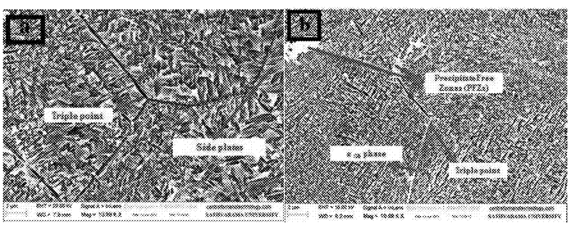
As can be seen from Figs. 3(b) and 3(c), there are some zones in SA 8 h/500 °C condition and DA 10 h/300 °C +10 h/500 °C condition, where precipitation of α has not occurred. No such α precipitate free zones (PFZs) were noticed in SA 10 h/500 °C condition and DA 24 h/250 °C +8 h/500 °C condition. Some PFZs are near or at the β grain boundaries, while some others are located well away from the grain boundaries. Such PFZs control the fatigue performance of the alloy, when heat treated to high strength levels [7]. It is thus necessary to monitor their presence in the microstructure and identify treatments which result in microstructures free from such zones. The amount and size of PFZs, it has been reported in case of β-C alloy, can be very sensitive to marginal variations in aging parameters [7].

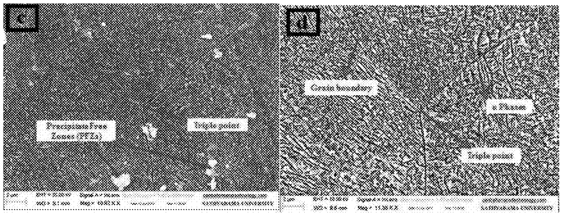
3.6.1.1 Hardness of PFZs Relative to the Surrounding Matrix

In order to get a further understanding of the nature of PFZs, micro Vickers hardness measurements were carried out on the 10 h/300 °C +10 h/500 °C DA specimens having PFZs in the microstructure. Comparative hardness evaluation of the PFZ (where there was no α precipitation) and the matrix surrounding it (where α phase precipitation has occurred) was carried out. The average hardness inside the PFZ was 370 HV; it was 405 HV in the surrounding matrix. In view of the smallness of the PFZs, it is possible that the surrounding matrix has also contributed to the measurements made in PFZs. But it clearly emerged that PFZs were significantly softer compared to the surrounding matrix. Similar observations were made by Schmidt et al for the case of beta titanium alloy Ti 38-644 (β-C) [7].

<u>3.6.2.2 Accounting for the Observations made in Section 3.6.1 Regarding Occurrence or Otherwise of PFZs</u>

An explanation of the occurrence of PFZs in 8 h/500 °C sample, but not in 10 h/500 °C sample will now be attempted. Breslauer and Rosen [8] studied precipitation in this very alloy after aging at 540 °C. They examined specimens with two different grain sizes – 35 and 120 μ. The material used for the present study is having a grain size closer to 35 μ. In the 35 μ specimen of Breslauer and Rosen [8], precipitation starts at grain boundaries and progresses into the interior with time of aging. To obtain an apparent homogenous distribution of precipitates, the alloy must be aged for at least 8 h. After heat treatment for 8 h, full aging is achieved. For shorter aging times, PFZs were observed in the microstructure. It appears that after 8 h at 500 °C, full aging has not happened in the present study and hence PFZs were seen in the microstructure. Aging for 10 h has resulted in full aging; precipitation has swept through the entire area of the grain, resulting in PFZs not appearing in the microstructure.
An explanation of occurrence of PFZs in 8 h/500 °C samples, but not in 24 h/250 °C +8 h/500 °C samples shall also be attempted. The 24 h treatment at 250 °C is expected to create nuclei all over the grain area and lead to early precipitation process uniformly over the entire grain area. The precipitation of α at 500 °C then occurs with the nuclei corresponding to the preprecipitation serving as precursors, and thus takes place uniformly without any PFZs in the microstructure. The 250 °C hold has resulted in acceleration of the precipitation process, in that in 8 h of hold at 500 °C, precipitation has swept through the entire grain area. Without 250 °C hold, there were PFZs after 8 h at 500 °C; it takes 10 h at 500 °C for the decomposition reaction to sweep through the entire area.
It remains to be explained as to why PFZs are seen in 10 h/300 °C +10 h/500 °C DA condition. It is possible that cluster like α phase precipitation occurred in a heterogeneous manner after 10 h at 300 °C and the regions adjoining these clusters have evolved as PFZs during second step aging. That α phase precipitates even at 300 °C has been demonstrated [9]. XRD patterns clearly show spectral lines corresponding to α phase after aging at 300 °C for 150 h [9]. It is not known, as to what are the sites which promoted heterogeneous nucleation of α at 300 °C. Further studies are required to get a complete understanding.





Figures 4. FESEM microstructures of selected SA and DA samples (a) SA at 10 h / 500 °C (b) SA at 8 h / 500 °C (c) DA at 8 h / 300 °C + 10 h / 500 °C (d) DA at 24 h / 250 °C + 8 h / 500 °C

Having discussed in detail the mechanical properties obtained in the two SA conditions and the two DA conditions and the corresponding microstructures observed at the SEM level, it is now to be examined as to which treatment is the best from the fatigue life point of view. PFZs were found to occur in the microstructure in 8 h/500 °C (SA) and 10 h/300 °C +10 h/500 °C (DA) conditions. They are expected to result in poor behaviour under fatigue loading conditions. No PFZs occurred in 10 h/500 °C (SA) and 24 h/250 °C +8 h/500 °C (DA) conditions; the latter condition has a higher strength with a comparable ductility and hence expected to be the best choice for fatigue loading condition

3.7 Fractography of Broken Tensile Test Pieces

Fractographic examination was carried out on broken tensile tests samples in SA and DA conditions. There was much of facetted fracture in both SA and DA samples, when aging temperatures were in the range 300-450 °C. Fracture surface of SA sample aged at 100 h / 300 °C showing nil ductility is shown in Fig. 5 (a). Material separation has occurred extensively by cleavage. The presence of ω phase in the microstructure is expected to lead to highly localized slip and early crack nucleation and propagation without macroscopic plastic deformation. There is a possibility that some amount of fracture has occurred along grain boundaries. In the high hardness condition associated with nil ductility, the hardness difference between the matrix and any grain boundary α present in the microstructure would be very high, leading to cracking along grain boundary. SA and DA involving aging at 500 °C resulted in substantial amount of fracture by microvoid coalescence. Fracture surface of DA sample (10 h / 300 °C + 10 h / 500 °C) is shown in Fig. 5 (b); the wide spread dimpled appearance is easy to notice.

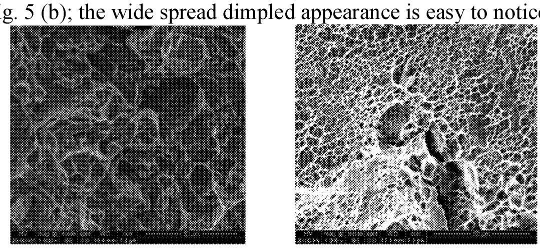


Figure 5. (a) Fracture surface after SA at (100 h / 300 °C), showing extensive cleavage fracture (b) Fracture surface after DA (10 h / 300 °C + 10 h / 500 °C) showing wide spread microvoid coalescence

4. Conclusions

1. For the titanium alloy Ti15-3, a relatively high aging temperature of 500 °C is to be adopted as part of SA or DA treatment to realize a good balance of strength and ductility. Two SA conditions (8 h/500 °C, 10 h/500 °C) and two DA conditions (24 h/250 °C +8 h/500 °C, 10 h/300 °C +10 h/500 °C) were accordingly studied in detail.
2. DA treatment results in higher strength compared to its counterpart SA treatment.
3. Precipitate free zones (PFZs) were found to occur in the microstructure in 8 h/500 °C (SA) and 10 h/300 °C +10 h/500 °C (DA) conditions. They are expected to result in poor behaviour under fatigue loading conditions.
4. No PFZs occurred in 10 h/500 °C (SA) and 24 h/250 °C +8 h/500 °C (DA) conditions; the latter condition has a higher strength with a comparable ductility and hence expected to be the best choice for fatigue loading conditions.
5. Aging in the temperature range 300-350 °C lead to near zero or nil-ductility with accompanying brittle fracture. Single/double aging involving 500 °C resulted in ductile fracture, with material separation occurring extensively through microvoid coalescence.

Acknowledgments
The work reported here has been carried out with the funds received under ER & IPR scheme of DRDO (Project number: ER & IPR number. ERIPR/ER/0903811/M/01/1250). The authors would like to thank DRDO for granting the project. The authors also like to thank the Management of VIT University, Vellore for their support and encouragement to publish this paper

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