

Grain Refinement by Means of Rapid Heating in Ti-2.5Cu and TIMETAL LCB

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The influence of thermomechanical treatments on microstructure development in the α titanium alloy Ti-2.5Cu and in the metastable β alloy TIMETAL LCB was studied. Conventional furnace heat treatments as well as rapid heat treatments through application of direct resistance heating were utilized to produce lamellar microstructures in Ti-2.5Cu as well as equiaxed microstructures in both Ti-2.5Cu and TIMETAL LCB. Rapid heat treatment as opposed to conventional furnace heat treatment led to much finer microstructures in both Ti-2.5Cu and TIMETAL LCB alloys. While the refinement in prior β grain size or colony size improved tensile ductility and LCF strength in lamellar Ti-2.5Cu, the reduction in α grain size in equiaxed Ti-2.5Cu and β grain size in TIMETAL LCB enhanced HCF strength.

Keywords: Rapid heating; microstructural refinement, fatigue performance, Ti-2.5Cu, TIMETAL LCB

1. Introduction

Thermomechanical treatments are often utilized to refine microstructures in the various classes of titanium alloys. For example, fine equiaxed or duplex (primary α in a lamellar ($\alpha+\beta$) matrix) microstructures in ($\alpha+\beta$) alloys such as Ti-6Al-4V can be generated from coarse grained β annealed material through ($\alpha+\beta$) working with subsequent subtransus heat treatments¹. Unfortunately, fully lamellar microstructures which are produced by supratransus heat treatments suffer from rapid grain growth in the single β phase field leading to large β grain sizes and upon cooling to corresponding large colony sizes typically of the order of a few hundred microns². These large colony sizes usually result in LCF behaviour of fully lamellar microstructures being inferior to duplex or fully equiaxed structures³. Earlier work on Ti-6Al-4V and VT-6 has shown that reducing the former β grain size by short time recrystallization in the β phase field applying rapid heating techniques can result in enhanced tensile ductilities and improved fatigue performance⁴. The present work was undertaken to study rapid heating effects on potential refinement of fully lamellar as well as fully equiaxed microstructures in Ti-2.5Cu and on the resulting tensile and fatigue properties. For comparison, rapid heating effects in a metastable β titanium alloy TIMETAL LCB were also included in this study.

2. Experimental procedures

The Ti-2.5Cu alloy was delivered as 10mm thick rolled plate. From this material, 10mm cylindrical bars were machined and annealed in the β phase field at 930°C for 30 min followed by water-quenching. This material was swaged at room temperature to 6.5mm diameter ($\phi = 0.9$). From this material, cylindrical blanks with a length of 60mm were taken and conventionally furnace annealed at either 805°C or 930°C for 1 hour followed by water-quenching. Other blanks were rapidly heat treated to these temperatures using direct resistance heating applying a voltage of about 5V and a current of 150A. The heating rate was roughly 100 Ks⁻¹. After reaching the desired temperatures, materials were water-quenched. Aging was

done by annealing material first at 400°C for 8 hours followed by annealing at 475°C for 8 hours.

The TIMETAL LCB alloy was delivered as 25mm cylindrical bar with a fine β grain size and primary α phase at the grain boundary triple points. Material was swaged at 660°C down to 6.7 mm ($\phi = 2.6$). From this material, cylindrical blanks with a length of 60mm were taken and conventionally annealed in the β phase field either at 805°C for 15 minutes or at 900°C for 1 hour. Other blanks were rapidly heated to 895°C followed by water-quenching. Aging was done by annealing at 540°C for 8 hours. Tensile tests of the various conditions were performed on threaded cylindrical specimens having gage lengths and diameters of 25 and 5mm, respectively. Young's moduli were measured by direct strain measurements with strain gages attached to the gage lengths of the specimens. Initial strain rates were $6.7 \times 10^{-4} \text{ s}^{-1}$. Fatigue tests on electrolytically polished hour-glass shaped specimens (100 μm removed from as-machined surface) were performed in rotating beam loading ($R = -1$) at a frequency of about 50 s⁻¹.

3. Results and Discussions

Microstructures of the various conditions in Ti-2.5Cu and TIMETAL LCB are illustrated in Figs. 1 and 2. After the β anneal, the as-received (AR) microstructure in Ti-2.5Cu having slightly elongated α grains (Fig. 1a) was transformed to a martensitic structure. After swaging at room temperature, the plastic bending of the martensite plates is clearly visible (Fig. 1b). Conventional furnace annealing at 805°C and 930°C led to recrystallized equiaxed (EQ) (Fig. 1c) and lamellar (L) (Fig. 1d) microstructures, respectively. Similar results are reported⁵. Utilizing rapid heating instead of conventional furnace heating resulted in fine equiaxed (FEQ) (Fig. 1e) and fine grained lamellar (FGL) (Fig. 1f) microstructures, respectively. Conventional β annealing at 900°C and 805°C of TIMETAL LCB previously swaged at 660°C resulted in coarse grain sized (CG) (Fig. 2a) and intermediate grain sized (IG) (Fig. 2b) microstructures, respectively. Rapid heating to 895°C led to a fine grained (FG) microstructure (Fig. 2c). After aging at 540C for 8h, these microstructures appear as illustrated in (Fig. 2 d-f).

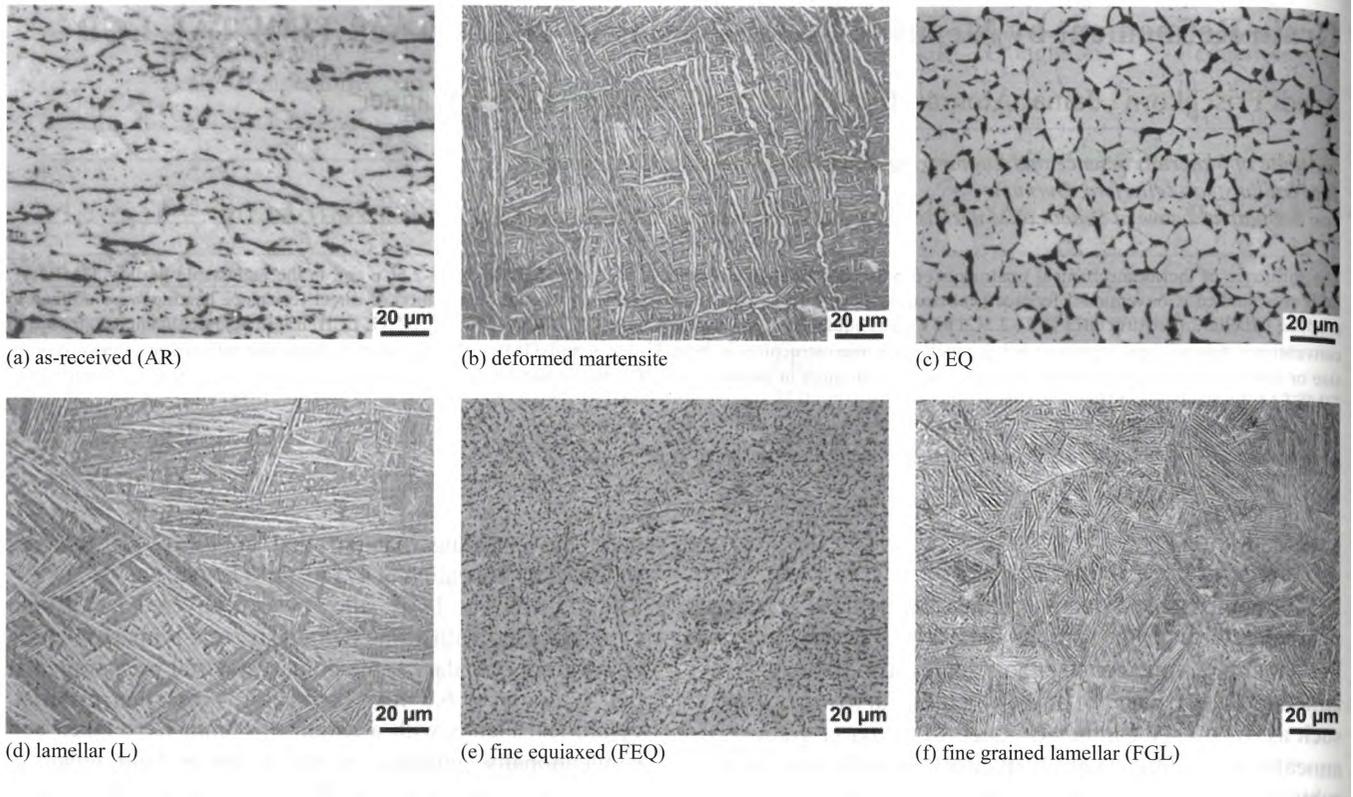


Figure 1. Microstructures of Ti-2.5Cu

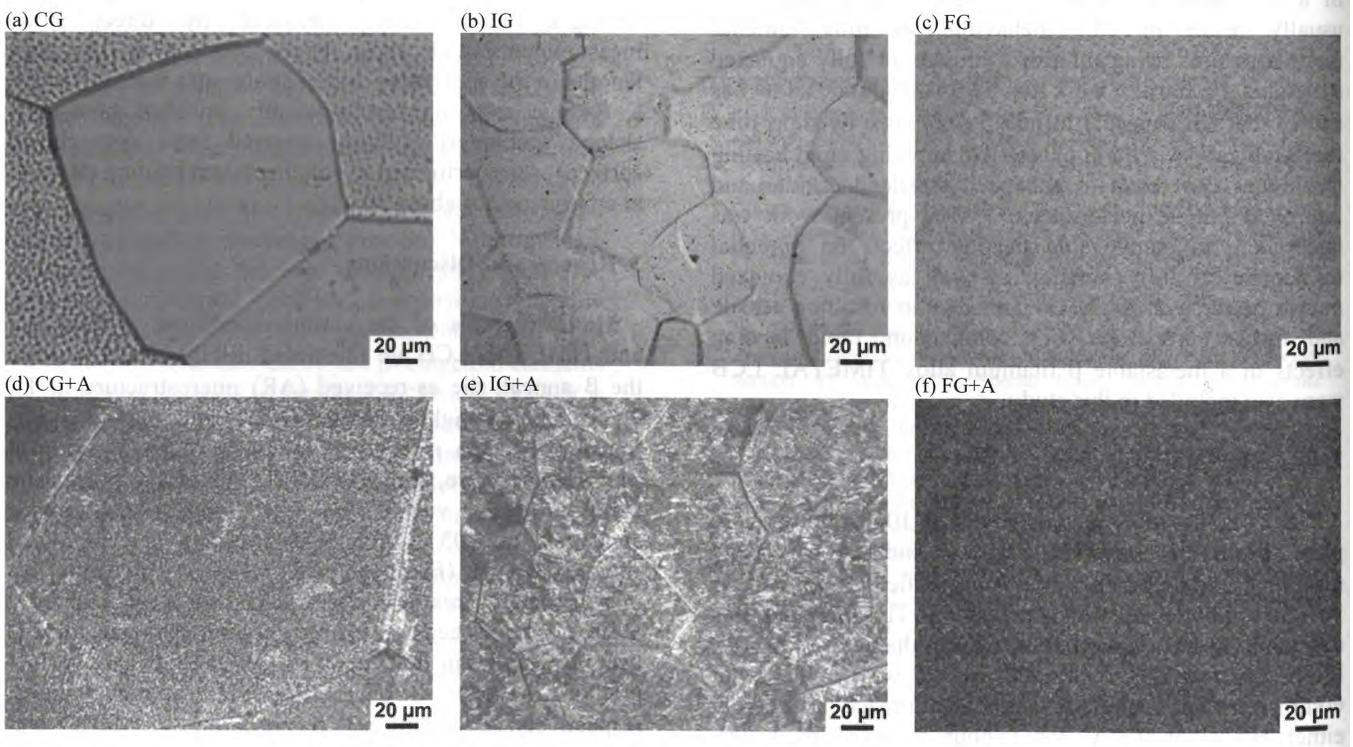


Figure 2. Microstructures of TIMETAL LCB

Tensile properties of the various conditions of Ti-2.5Cu are listed in Table 1.

Table 1. Tensile properties of Ti-2.5Cu (A = 8h 400°C + 8h 475°C)

Micro-structure	Grain Size (μm)	E (GPa)	σ_Y (MPa)	UTS (MPa)	EI (%)	ϵ_F
EQ	15	102	520	715	18.6	0.52
EQ+A	15	108	550	690	18.9	0.54
FEQ	3	99	545	695	15.8	0.58
FEQ+A	3	104	600	735	13.5	0.50
L	300 ^x	110	615	710	15.9	0.29
L+A	300 ^x	110	730	810	12.7	0.32
FGL	50 ^x	101	600	720	17.4	0.61
FGL+A	50 ^x	108	755	800	9.6	0.47

^x colony size

Aging of the equiaxed microstructures increases yield stress by 30 MPa and 55 MPa for the conventionally heat treated (EQ) and rapidly heated (FEQ) microstructures, respectively (Table 1). No pronounced effect of aging was observed with regard to tensile ductility after conventional heat treatment while the rapidly heated material exhibits a mild decrease in ductility. A reduction in α grain size from 15 to 5 μm increases yield stress by 25 MPa and 50 MPa for the unaged and aged materials, respectively. As opposed to yield stress, this grain refinement hardly affects tensile ductility.

Compared to the equiaxed microstructures, yield stress values in the lamellar microstructures are significantly higher, particularly, after aging. No effect of colony size on yield stress or UTS is observed since these properties are mainly affected by the widths of the α lamellae⁶⁻⁸⁾ which were kept constant by using the same cooling rate. As expected, tensile ductilities in the lamellar microstructures are markedly lower than in the equiaxed structures. Similarly to the equiaxed microstructures, aging does not reduce the tensile ductilities of the conventionally heat treated lamellar microstructures while some loss in ductility is seen in the rapidly heated material. As expected from previous results on ($\alpha+\beta$)⁴⁾ and near- α titanium alloys⁹⁾, the tensile ductilities are much higher in the fine colony sized material.

Tensile properties of the various conditions of TIMETAL LCB are illustrated in Table 2.

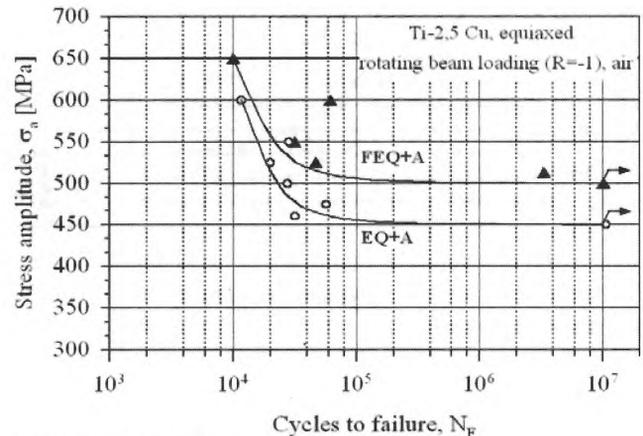
Table 2. Tensile properties of TIMETAL LCB

Micro-structure	Grain Size (μm)	E (GPa)	σ_Y (MPa)	UTS (MPa)	EI (%)	ϵ_F
CQ	150	70	1025	1035	13.3	0.75
CG+A	150	109	1300	1310	0.38	0.02
IG	70	82	1120	1120	16.9	0.77
IG+A	70	114	1310	1355	4.1	0.12
FG	2	78	1130	1140	18.0	0.90
FG+A	2	113	1315	1380	11.2	0.53

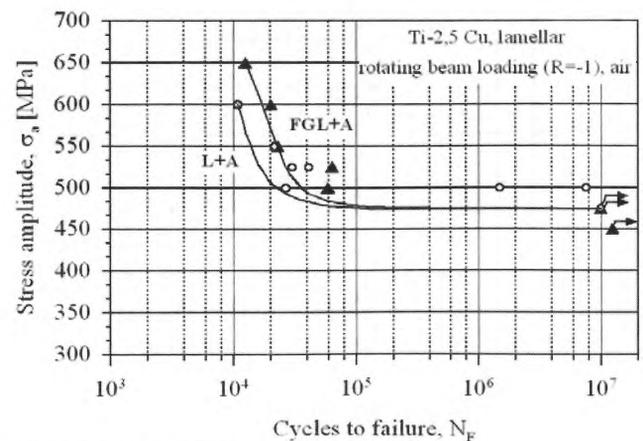
Aging by precipitation of fine secondary α particles in the β matrix of CG is seen to increase Young's modulus by as much as 30 GPa and yield stress by almost 300 MPa. This aging response in CG is somewhat more pronounced than in IG and FG (Table 2).

While a reduction in grain size from 150 to 70 μm increases the yield stress from 1025 to 1120 MPa in the unaged conditions, no additional increase in yield stress is seen by further refining the grain size from 70 to 2 μm . After aging, the yield stress is fully independent of grain size. As opposed to yield stress, UTS values clearly increase with a decrease in grain size. Whereas the reduction in grain size from 150 to 2 μm only slightly increases the tensile ductility in the unaged microstructure from 0.75 to 0.90, the beneficial effect of grain refinement is much more pronounced in the aged conditions. The same grain refinement leads to an increase in tensile ductility from 0.02 to 0.53. Thus, the fine grained age-hardened microstructure combines a UTS of almost 1400 MPa with this high tensile ductility of 0.53 (Table 2).

The S-N curves of the various age-hardened microstructures in Ti-2.5Cu are shown in Fig. 3 comparing grain size effects in the equiaxed (Fig. 3a) and lamellar microstructures (Fig. 3b).



a) Equiaxed microstructures



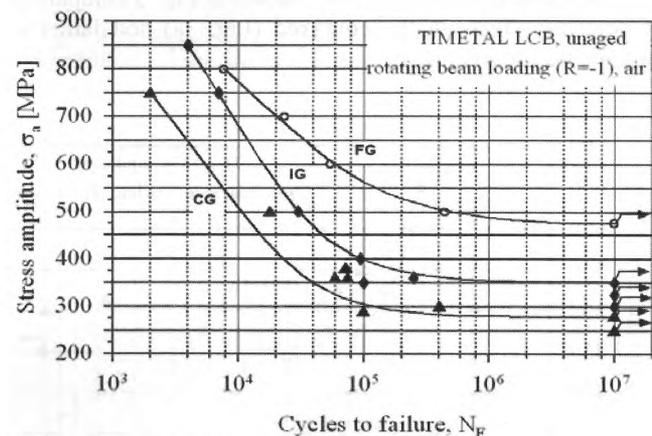
b) Lamellar microstructures

Figure 3: S-N curves in Ti-2.5Cu

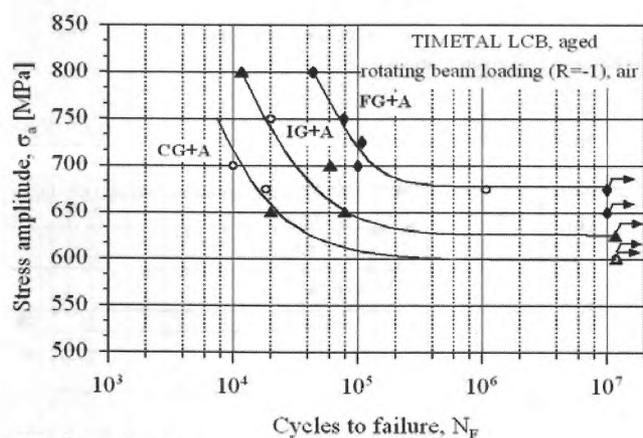
A refinement in α grain size from 15 to 5 μm in the equiaxed microstructures improves both LCF and HCF performance (Fig. 3a). This improvement can be derived from the 50 MPa higher yield stress of FEQ compared to EQ (Table 1) which results in a higher resistance to fatigue crack nucleation.

As seen in Fig. 3b, a refinement in the colony size from 300 to 50 μm in the lamellar microstructures leads to a fatigue life improvement in the LCF regime without an improvement in HCF strength. The enhanced fatigue life at high stress amplitudes of FGL as compared to L can be derived from the higher tensile ductility in FGL amounting to 0.47 as opposed to 0.32 in L. An increase in tensile ductility is known to result in lower crack growth rates of microcracks in this material thus, in a fatigue life improvement⁹⁾.

The S-N curves of the various microstructures in TIMETAL LCB are shown in Fig. 4 comparing grain size effects in the unaged (Fig. 4a) and aged microstructures (Fig. 4b).



a) Unaged microstructures



b) Aged microstructures

Figure 4: S-N curves in TIMETAL LCB

There is a significant enhancement in fatigue performance by refining the grain size in unaged TIMETAL LCB (Fig. 4a). A decrease in grain size from

150 μm over 70 μm to 2 μm increases the 10^7 cycles fatigue strength from 275 MPa over 350 MPa to 475 MPa. Note the very marked fatigue strength increase from 350 MPa to 475 MPa by decreasing the grain size from 70 μm to 2 μm . Since this grain refinement does not result in a concomitant increase in yield stress (Table 2), the enhancement in fatigue strength by 125 MPa is thought to be caused by the refinement in microstructural unit size, e. g. finer slip distribution which also leads to an increase in tensile ductility from 0.77 to 0.90 (Table 2).

Aging is seen to drastically improve the fatigue strength of the various grain sized conditions of TIMETAL LCB (Fig. 4b, compare with Fig. 4a). Furthermore, the degree of improvement clearly depends on grain size. With a decrease in β grain size from 150 μm over 70 μm to 2 μm , this improvement declines from 325 MPa over 275 MPa to 200 MPa (compare Fig. 4b with Fig. 4a).

Calculating the ratio $\sigma_a 10^7 / \sigma_Y$ of the various grain sized and age-hardened conditions, this value increases from 0.27 over 0.31 to 0.42 in the unaged material if the grain size is reduced from 150 μm over 70 μm to 2 μm . After aging, this ratio increases to 0.46, 0.48 and 0.51 for the grain sizes of 150 μm , 70 μm and 2 μm , respectively.

In a recent publication on aged TIMETAL LCB, fatigue crack nucleation was found to be associated with α precipitates along β grain boundaries¹⁰⁾. To determine if this is also the case in the present work on TIMETAL LCB, fatigue crack nucleation sites and microcrack growth paths in the various microstructures will be studied in future work.

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REFERENCES

- 1) G. Lütjering : Mat. Sci. Eng., A **243** (1998) pp. 32.
- 2) S. Saal, L. Wagner, G. Lütjering, H. Pillhoefer, M. A. Daeubler: Z. Metallkde. **81**, (1990) pp. 535.
- 3) L. Wagner, G. Lütjering: Z. Metallkde. **87** (1987) pp. 369.
- 4) O. M. Ivasishin, P. E. Markovski, L. Wagner and G. Lütjering: *Titanium Products and Applications*, TDA (1990), pp. 99.
- 5) K.-H. Kramer, R. Arndt, J. Budde: Metall, Heft **10**, 27. Jhg. (1973) pp 983.
- 6) R.I. Jaffee, L. Wagner, G. Lütjering: Les Edition de Physique, 1988, pp. 1501.
- 7) A. Styczynski, L. Wagner, C. Mueller, H. E. Exner: *Microstructure/property relationships of titanium alloys*, TMS, Warrendale, (1994), pp. 83.
- 8) A. Berg, J. Kiese, L. Wagner: *Light-weight alloys for aerospace applications III*, TMS, Warrendale (1995) pp. 407.
- 9) J. K. Gregory and L. Wagner: *Fatigue 90* (H. Kitagawa and T. Tanaka, eds.) MCEP (1990) pp. 191.
- 10) B. Y. Kokuoz, Y. Kosaka, H. J. Rack: J. Mat. Eng. and Performance, **14**, no. 6 (2005) pp. 773.