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# Effects of lanthanum on structures and grain sizes of Ti-44 Al alloy<sup>©</sup>

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Abstract: By vacuum arc melting, lanthanum (La) was added in the range from 0.05 % to 0.3 % (mole fraction) to binary Ti-44 % Al alloy to refine its fully lamellar (FL) colonies for high ductility. Effects of La on structures and grain sizes of the alloy were determined. It was observed that La greatly promoted growth of columnar grains in as cast structures of the ingots and reduced their diameters. The ingots were subsequently heat treated to obtain well-defined fully lamellar structures. The material without La displayed large average size of equiaxed lamellar colonies over 1 400  $\mu$ m (resulted from five measurement positions in a sample), and the material with 0.1 % addition of La consisted of the finest average lamellar colonies as 381  $\mu$ m, the finest grain size was 337  $\mu$ m at top position. When La addition was over 0.1 %, La stabilized the columnar structure and it was difficult to refine the columnar grains in heat treatment, therefore, grains were coarser.

Key words: titaniu m-alu miruiu m alloy; lanthanu m; addition structures Document code: A

#### 1 INTRODUCTION

TiAl based intermetallic alloys have been expected as a new kind of aircraft and aerospace material to substitute for Ni<sub>3</sub> Al and Fe<sub>3</sub> Al alloys owing to their high toughness at elevated temperature and low density[1~3]. A great number of results from other researchers show that microstructure is the major factor affecting mechanical properties of the alloy [4~10]. It is stated that fine FL structure grains perform an outstanding balance between fracture toughness and creep resistance at elevated temperature and ductility and strength at room temperature. In recent years, an untired effort to refine the FL structure grains has being carried out. For example, the colony sizes after isothermal forging would still be at least  $500 \sim 1000 \,\mu \,\text{m}^{[7]}$  and the colony sizes after reciprocating hot mechanical process were as fine as  $20 \sim 30 \mu \, m^{[11]}$ . The alloying process to refine FL structure colonies for improving ductility at room temperature was recently another main research topic. [12~14] The grain sizes were reduced to 100  $\mu$ m  $b_V$  adding 0.8% boron into the alloy<sup>[15]</sup>.

Rare earth elements (RE) have been used for many years to purify and refine the microstructures of such materials as steels, cast irons, aluminium alloys, titanium alloys and magnesium alloys<sup>[16,17]</sup>. It is reported that rare earth element, Er, could change the behavior of solidification of TiAl alloy[18] and La could improve the ductility of Ti Al alloy at room temperature<sup>[19]</sup>. However, the use of REs in refining Ti Al based alloys and their effects on mechanical properties have not been seriously investigated. In the present study RE element La was added to Ti-44 % Al alloys to refine the la mellar colonies in the fully lamellar structured materials. The effects of La on the macro and micro structures and the grain sizes in casting ingots of the alloys are presented here.

## 2 EXPERIMENTAL MATERIALS AND PROCEDURES

#### 2.1 Preparation of samples

The raw materials of pure titanium, alu-

minum and lanthanum total weighted to 30 g were melt in a vacuum arc melting furnace and cast into an ingot with 23 mm in length, 19 mm in width and 19 mm in height. The purities of the metals used were as follows: 99.9 % for Ti, 99.99 % for Al, and 99.5 % for La. The a mounts of La addition were 0.05 %, 0.1 %, 0.2 %, and 0.3 %. The results of che mical analysis for Ti 44 Al-0.2 La alloy were 56.41 % for Ti, 43.40 % for Al and 0.19 % for La; and those for Ti 44 Al-0.3 La alloy were 56.29 % for Ti, 43.43 % for Al and 0.28 % for La, which indicated that actual compositions were fairly close to the nominal ones.

The ingots were cut in the middle along the height and length directions. Half of the ingot cross section examined and the five locations where samples were taken for grain size measure ments are shown in Fig.1.

Samples of cast ingots were wrapped with Ta foil and sealed in quartz tubes filled with Ar for heat treatment. The sealed samples were heated and kept at 1350 °C in the single  $\alpha$  region for 1 h to cause the recrystallization of  $\alpha$  grains. This was followed by a controlled furnace cooling at 6 °C/ min to 1000 °C and then at 1.85 °C/ min to room temperature.

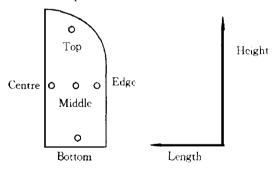


Fig.1 Sketch of positions checked in length-height section

### 2.2 Processes of observations of structures and measurements of grain sizes

Samples from different locations were prepared for examination of both macro and micro structures. The standard procedures of metallography were followed in polishing samples. The macrostructures were taken  $b_{\rm Y}$  a normal camera

and the microstructures were observed by optical microscopy. The oxides and aluminides of La were observed by scanning electron microscopy (SEM) and their compositions were analyzed by electron spectrometer and pulsed NMR spectrometer. The lamellar colony sizes were measured using the circular intercept procedures as described in ASTM standard El12-95.

#### 3 EXPERIMENTAL RESULTS

#### 3.1 Effects of La on structure of TiAl allov

#### 3.1.1 Effects of La on as-cast structure

Effect of La on as-cast macrostructure of the alloy is shown in Fig.2. A surface thin layer of fine, equiaxed grains was observed under microscopy in all three materials. The columnar grains can be seen to grow from the surface layer to the center in the opposite direction of heat flow, and the size of the columnar zone in the vertical direction is as small as 2 mm in the binary alloy (without La) and the sizes in the alloys with 0.05 % and 0.3 % La are about 7 mm and 10 mm, respectively. It is seen from Fig.2 that La has refined the equiaxed colonies in the center of the ingots.

Effects of La on microstructure of the ingots are shown in Fig. 3. The diameters of the columnar grains were reduced from  $150 \sim 200 \, \mu \, m$  in the material without La to about  $50 \, \mu \, m$  in the material with  $0.3 \, \%$  La. So, it is known that La not only promotes the growth length but also reduces the diameters of the columnar grains. The grains in the top of the ingot are equiaxed lamellar colonies .

### 3.1.2 Effects of La on structure after being heat treated

The macrostructures of the binary alloy and the alloys with La additions after the heat treatment are shown in Fig.4, from which it is quite obvious that the columnar colonies were disappeared and equiaxed grain structures were observed in the binary alloy and the alloys with La additions: coarse in the binary alloy and much refined in the alloys with La. For the alloy with 0.1% La, the finest and most uniform grains were observed. While addition amount was over 0.1%, La enhanced the stabilization of the

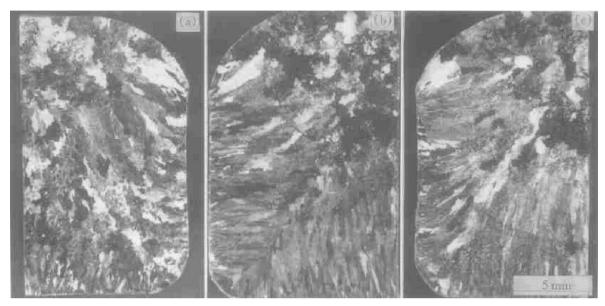
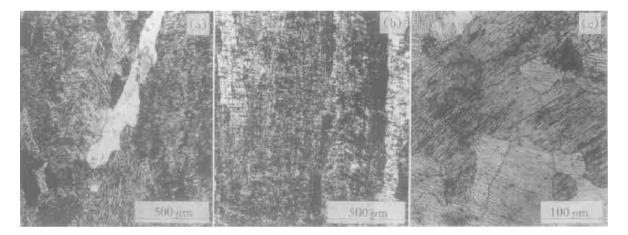


Fig.2 Effects of La on macrostructure of cast ingots (a) -No La; (b) -0.05% La; (c) -0.3% La



**Fig.3** Effects of La on microstructure of casting ingots (a) - No La , bottom ; (b) -0 .3 % La , bottom ; (c) -0 .05 % La , top

columnar colonies after heat treatment and some reminiscence of the columnar grains could still be seen near the bottom of the ingots. On the other hand, the grain sizes of ingot with 0.05 % La were refined greatly compared with the binary alloy but were coarser than those of the alloy with 0.1 % La. These mean that 0.1 % of La is the proper addition for refining grain sizes in this experiment condition.

Fig. 5 shows the microstructures at the bot-

to m of the ingots after being heat treated. The fully lamellar structure was seen in the ingot with  $0.05\,\%$  La from Fig.5(a), the columnar colonies had been broken to lumps in the ingot with  $0.2\,\%$  La from Fig.5(b), and in decomposed fine columnar grains which were half tone, were observed in the ingot with  $0.3\,\%$  La from Fig.5(c). It is concluded that La had drastically stabilized the columnar grains in heat treatment processing.

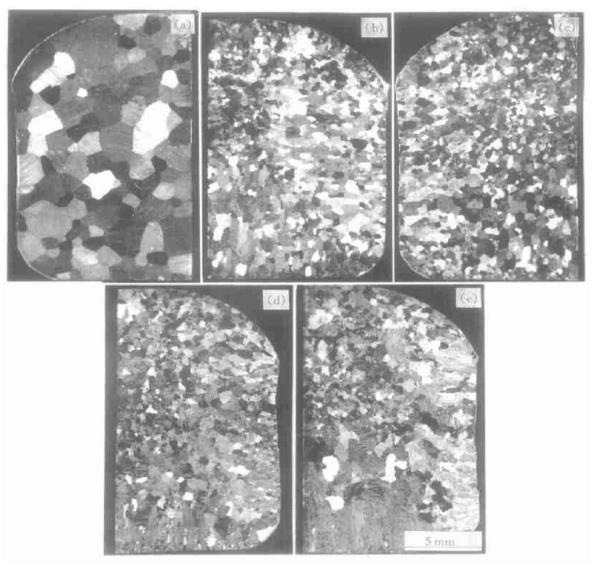


Fig. 4 Effects of La on macrostructure of ingots after being heat treated (a) -No La; (b) -0.05% La; (c) -0.1% La; (d) -0.2% La; (e) -0.3% La

### 3.2 Appearances and distributions of oxides and aluminides of La

Amounts of oxides and aluminides of La were raised as La additions increased. From Fig.6(a), very fine oxides were seen to appear in the matrix in the ingot with  $0.1\,\%$  La and some La aluminides lay along the heat treatment boundaries to be seen in Fig.6 (b) as La addition was  $0.3\,\%$ . Fig.6(c) shows the appearance of coexistence of an oxide and an aluminide.

Compositions of the oxides and aluminides

analyzed are listed in Table 1 , from which it is deduced that La oxides are  $\text{La}\,O_2$  and  $\text{La}_2\,O_3$  , and La aluminides are  $\text{La}_2\,Al_3$  and  $\text{La}\,Al_3$ .

The characteristics of appearances and distributions of the oxides and the aluminides show that oxides of La formed in liquid and at the period of solidification of the alloys and aluminides of La formed as the temperature was dropped down during heat treatment. La atoms over saturated in grains diffused toward boundaries or oxides in the grain and reacted with aluminum

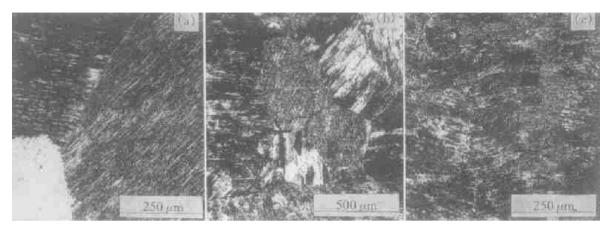


Fig. 5 Effects of La on microstructures of ingots after being heat treated at bottom
(a) -0.05 % La;(b) -0.2 % La;(c) -0.3 % La

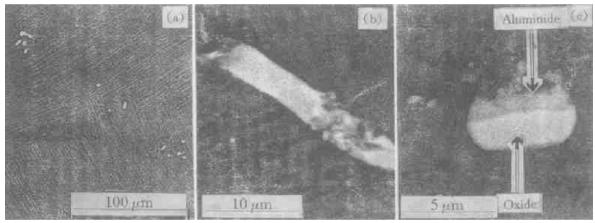


Fig.6 Appearances and distributions of oxides and aluminides
(a) —Oxides in matrix, 0.1 % La; (b) —Aluminide in a boundary, 0.3 % La;
(c) —Co-existence of oxide and aluminide, 0.3 % La

**Table 1** Results of compositions analysis of La oxides and aluminides (mole fraction, %)

Sa mple	Ti	Al	О	La	Phase
Ti- 44 Al- 0 .1 La	4.78	0.62	57.62	36 .98	Oxide
Ti- 44 Al- 0 .3 La		0.28	56.87	42 .85	Oxide
Ti- 44 Al- 0 .3 La		4 .61	73 .60	21 .79	Oxide
Ti- 44 Al- 0 .3 La		62.13		37.87	Aluminide
Ti- 44 Al- 0 .3 La		65 . 23		34.77	Aluminide
Ti- 44 Al- 0 .3 La		74.61		25.39	Aluminide

atoms to form aluminides as soon as certain concentration of La atoms was reached.

#### 3.3 Effects of La on grain size

The lamellar colony sizes were measured for

materials with different La contents at the five locations illustrated in Fig .1, and the results are shown in Fig .7.

It can be seen that the addition of 0.1 % La produced the finest colonies and the most uniform distribution of colony sizes in most positions of the ingot and the local finest colonies at the top position of the ingot was as fine as  $337\,\mu\,m$ . The colony sizes in the ingot with 0.05 % La were slightly smaller than those of ingot with 0.1 % La at the bottom and middle, however, the colony sizes at other positions were larger than those of ingot with 0.1 % La . The colony sizes are in a tendency of increase in all positions

when La additions are over 0.1 %. The reason for this are: (1) La has promoted growth of columnar colonies in the as-cast structure and stabilized columnar colonies during heat treatment, and (2) the columnars are liable to merge together in radial direction of them to form coarse columnars in elevated temperatures because of their very small differences of lattice orientations between columnars [20].

Fig.7 Effects of La on grain size in different positions in ingots

The average colony sizes in the materials without La and with different additions of La are 1 424  $\mu$  m( no La) , 397  $\mu$  m ( 0.05 %La) , 381  $\mu$  m ( 0.1 % La) , 479  $\mu$  m ( 0.2 % La) , and 617  $\mu$  m ( 0.3 %La) , respectively .

#### 4 DISCUSSION

It has been shown in this investigation that La reacted initially with oxygen in Ti-44 % Al melt and then existed as free atoms to change solidification behavior of the alloy. It has been found that the temperature should be raised for breaking up the columnar grains when during heat treatment. This is an evidence of existence of La atoms in the lattice of the alloy. Meanwhile, the facts that the aluminides of La existed in boundaries and co-existed with oxides of La verify that atoms in grains migrated toward boundaries and oxides in lattice, because there

were many voids around lattices, and combined with aluminum to form aluminides at heat treatment temperature.

#### REFERENCES

- 1 Froes F H. Journal of Materials Science, 1992, 27: 5113.
- Whang S H. High Temperature Aluminides and Intermetallics. The Minerials, Metals, and Materials Society, 1990:465.
- 3 Kim Y-W. JOM, 1991, 43:40.
- 4 Kim Y-W. Acta Metallurgical Materials, 1992, 40 (6):1121.
- 5 Chan K S. Acta Metallurgical Materials, 1995, 43 (2): 439.
- 6 Darola R. Structural Intermetallics. The Minerials, Metals, and Materials Society, 1993: 143.
- 7 Kim Y W. Mater Sci Eng, 1995, Al 92/193: 519.
- 8 Zhou Kechao and Hang Baiyun. The Chinese Journal of Nonferrous Metals, (in Chinese), 1996, 6(3): 111.
- 9 He Yuehui and Huang Baiyun. The Chinese Journal of Nonferrous Metals, (in Chinese), 1997, 7 (1): 75.
- 10 Liu Wensheng and Huang Baiyun. The Chinese Journal of Nonferrous Metals, (in Chinese), 1997, 7 (4): 115.
- 11 He Yuehui. Materials Science and Engineering, (in Chinese), 1996, 14 (1): 35.
- 12 Daniel S. In: Proceedings of Materials Research Society Symposium, 1995, 364: 787.
- 13 Zhang W J. Materials Science and Engineering, 1990, Al 20:15.
- 14 Peng Chaoqun and Huang Baiyun. The Chinese Journal of Nonferrous Metals, (in Chinese), 1998, 8 (Supl. 1): 11.
- 15 Kim Y-W, Wagner R and Yamaguchi M. Gamma Titanium Aluminides. Warrendale, TMS, 1995: 637.
- 16 Guptu C K and Krishnan T S. Materials Science Forum: Trans Tech Publication, 1988: 89.
- 17 The Materials Information Society. Metals Handbook, 1990, 2: 720.
- 18 Bassler B T. In: Proc Mat Res Soc Symp, 1995, 364:1011.
- 19 Chen Shiqi. Acta Metallurgica Sinica, (in Chinese), 1994, 30 (1): A20.
- Ple mings M C. Solidification Processing. McGraw-Hill, Inc, 1974: 159.

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