Temperature Gradient Estimation of Floating Zone Furnace by Mean Lamellar Spacing Measurement in DS-processed TiAl Alloys

I. S. Jung*, Jong-Moon Park**, D. M. Wee*, Myung-Hoon Oh**,†

[1]*Department of Materials Science and Engineering, KAIST, Daejeon 305-701, Korea*
[2]*School of Materials Science and Engineering, Kumoh National Institute of Technology (KIT), Gumi,
Gyeongbuk 730-701, Korea*

Abstract   In this study, the temperature gradient of a floating zone (FZ) apparatus, which is very difficult to directly measure, could be estimated by using the relationships between the lamellar spacing of directionally solidified TiAl alloys and the solidification rate, It was found that the calculated temperature gradient of the FZ apparatus was much higher, and almost ten times higher than that of a Bridgman type directional solidification apparatus.

1. Introduction

The floating zone (FZ) technique is a commonly used method for growing single crystals of metals including intermetallic compound with high melting temperature, and it is also used as a directional solidification (DS) tool to control the orientation of microstructure in some alloy systems [1-3]. Recently, there are a lot of reports on TiAl-X(X is beta stabilizing elements, such as W, Mo and Nb et. al) DS-processed alloys and related mechanical properties using both FZ and Bridgman type [4-8]. From this point of view, in order to analyze the relationships between the microstructure of DS ingots and solidification rate during DS process, we have to know the temperature gradient of DS apparatus because the solidification rate (R) is determined by the multiple of the growth rate (V) and the temperature gradient (G) of the DS apparatus [9]. While the growth rate can be controlled by selecting the ratio of step motors in the control panel of DS equipment during the DS process, it is very difficult to change the tem-

perature gradient because it depends on the apparatus type itself.

The temperature gradient of a Bridgman type DS apparatus can be easily measured during the DS procedure, as shown in Fig. 1. However, as far as a FZ apparatus is concerned, it is very difficult to directly measure the temperature gradient during the DS procedure because ingots are produced without crucible and DS proceeds in an airtight quartz-tube with argon atmosphere. Therefore, the temperature gradient of FZ furnace could be numerically calculated by a computer simulation using heat transfer equations and thermodynamic data. Also, it could be changed by the alloy systems, flow rate of inert gas, the size of ingot etc., even the same equipment. Except the computer simulation, there have been no research results on the experimental measurement of the temperature gradient in FZ furnace using metallic systems. Therefore, in this study, the temperature gradient of a FZ furnace, which is thought to be one of the most important factors in assessing solidification behavior in the DS process, was estimated by the new idea on the basis

(a)

1: feeder
2: liquid zone
3: new grown crystal
4: supporter
5: halogen lamp

(b)
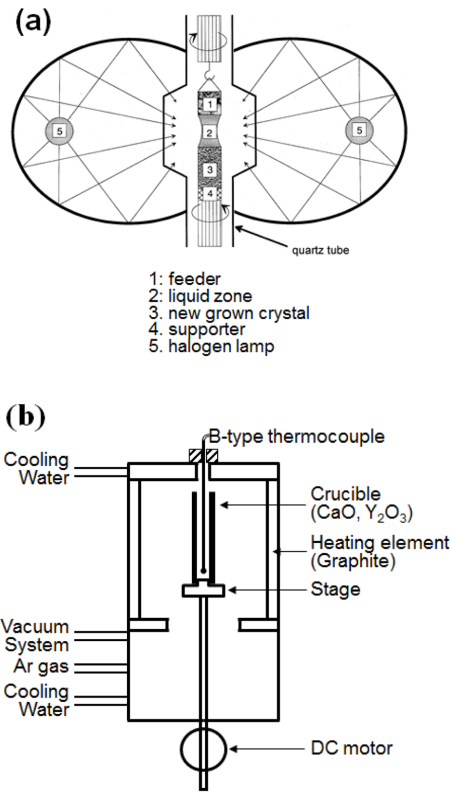
Fig. 1. Schematics of the (a) floating zone type and (b) Bridgman type DS apparatus used in this study.

of the following reports.

J. Tang [10], Y-W. Kim [11] and H. Umeda[12], as well as other studies, have reported that the lamellar spacing (L) of fully lamellar TiAl alloy is the function of solidification rate (R) and Al composition in TiAl alloys. In other words, the lamellar spacing of fully lamellar TiAl alloy is related only to the solidification rate, R, in a constant composition of TiAl alloy. In addition, they reported that lamellar spacing L is linearly related to the inverse square root of the solidification rate, $R^{-1/2}$, as shown in Fig. 2 [11].

In this study, above relationships were basically used in order to estimate the temperature gradient of an FZ apparatus. In the constant alloy composition of Ti-47Al-2W (at.%), it was thought that the lamellar spacing would be related only to the

temperature gradient and to the growth rate in the DS process. For the reference of the relationship between the lamellar spacing of Ti-47Al-2W alloy and the solidification rate, the temperature gradient of a Bridgman type apparatus was directly measured during the DS procedure. DS ingots were manufactured at various growth rates by using the Bridgman and FZ equipment. Based on the results obtained, the temperature gradient of a FZ apparatus was estimated using the relationships mentioned above.

2. Experimental Procedures

The alloy ingots were prepared as 20 g button shapes by using arc-melting furnace under argon gas atmosphere using pure elements (99.99 wt.% Ti sponge, 99.99 wt.% Al shot and 99.9 wt.% W powder). They were then re-melted to form rod shaped ingots (15 mm diameter and 90 mm length).

When using the Bridgman type DS apparatus showed in Fig. 1(b), the furnace temperature was increased to 1200°C maintaining vacuum at a 10^{-3} torr level. Then, the temperature was increased to 1600°C with an Ar gas atmosphere to protect air inflow from the outside. The furnace temperature was held at 1600°C for 30 min., and then the water-cooled stage descended at growth rates of 90 mm/h and 270 mm/h, respectively. The DS experiments were carried out using a Y_2O_3 crucible. The temperature gradient of the Bridgman type apparatus was directly measured during directional solidification by inserting a B-type thermocouple into the melt, as shown in Fig. 1(b). DS was also performed in an ASCAL FZ-SS35W optical floating zone (FZ) furnace [2] using growth rates of 30 mm/h and 90 mm/h, respectively, as shown in Fig. 1(a).

For measuring the lamellar spacing of the directionally solidified TiAl alloys, transmission electron microscopy (TEM) observation was performed. The specimens for TEM observation were

electro-polished in a solution of 30 ml perchloric acid + 175 ml *n*-buthanol + 300 ml methanol at −40 °C with a twinjet polisher.

3. Results & Discussion

In a directional solidification(DS) process, solidification rate (R, °C/min) is determined by the multiple of growth rate(V, cm/min) during the DS procedure

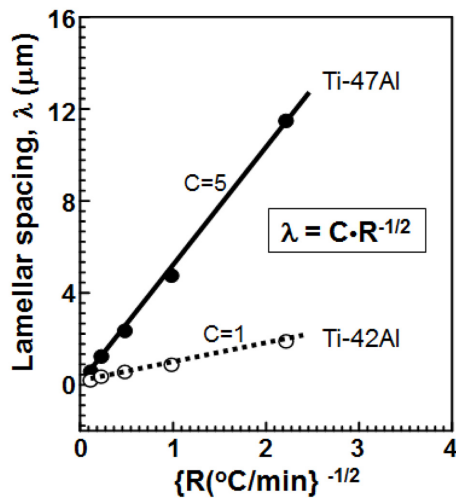**Fig. 2.** The linear relationship between the lamellar spacings (λ) and the inverse square root of the solidification rate (R^{-1/2}) [11].

and the temperature gradient(G, °C/cm) of the DS apparatus according to the following formula (1)[9]:

$$R\ (^{\circ}C/min) = V\ (cm/min) \times G\ (^{\circ}C/cm) \qquad (1)$$

It was also reported that the lamellar spacing (λ) of fully lamellar TiAl alloys is related to both alloy composition and solidification rate, R. If the TiAl alloy composition is fixed, the values of λ (lamellar spacing) have a linear relationship with the values of R^{-1} or $R^{-1/2}$ [5, 6]. In this study, the temperature gradient of a floating zone (FZ) type DS apparatus was simply estimated by using the relationship between the lamellar spacing and the solidification rate as explained above. At first, the Ti-47at.%Al-2at.%W DS alloys were manufactured by using a Bridgman type apparatus at growth rates of 90 and 270 mm/h. For the measurement of the lamellar spacing in the DS alloys, the fully lamellar microstructure of DS ingots were observed by TEM, as shown in Figs. 3(a) and (b). The γ/γ average lamellar spacing was assessed in more than 15 specimens of each DS ingot to ensure the reliability of the measurement; the results are listed in Table 1 with the specimen names and DS conditions, respectively. By using the FZ apparatus,
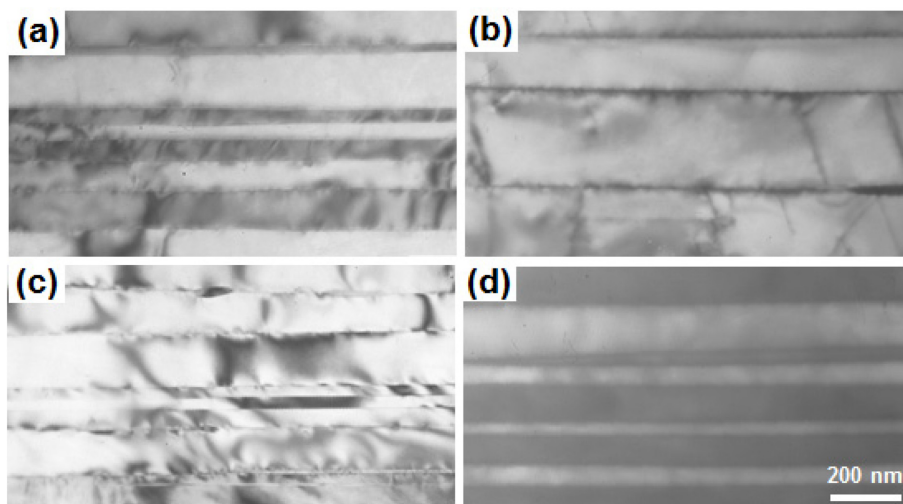

Fig. 3. TEM micrographs of DS TiAl alloys at the growth rates of (a) 270 mm/h, (b) 90 mm/h, (c) 90 mm/h and (d) 30 mm/h by using (a) and (b) a Bridgman type, (c) and (d) a floating zone type DS apparatus, respectively.

Table 1. Specimen names, DS conditions, growth rates and mean lamellar spacing of each DS alloy

Specimen name	Alloy composition	DS apparatus	Growth rate	Mean lamellar spacing
270B	Ti-47Al-2W	Bridgman	270 mm/h	124 nm
90B	Ti-47Al-2W	Bridgman	90 mm/h	212 nm
90F	Ti-47Al-2W	Floating zone	90 mm/h	77 nm
30F	Ti-47Al-2W	Floating zone	30 mm/h	125 nm

**Table 2.** Estimated temperature gradient of the floating zone type DS apparatus used in this study.

Specimen name	Solidification rate	Temperature gradient
270B	14.4°C/min	*32°C/cm
90B	4.8°C/min	*32°C/cm
90F	†38.8°C/min	†259°C/cm
30F	†14.2°C/min	†283°C/cm

*directly measured during directional solidification, †calculated in this study

DS alloys with the same composition were produced at growth rates of 30 and 90 mm/h. Figs. 3 (c) and (d) also showed the typical lamellar microstructures of FZ ingots and the mean lamellar spacing are also listed in Table 1.

In order to determine the relationships between the solidification rate and the lamellar spacing of TiAl alloys in DS process, we need to know the temperature gradient of the DS apparatus. Then, as the first step, the temperature gradient of the Bridgman type apparatus was directly measured by a B-type thermocouple during the DS procedure, and it was found to be approximately 32°C/cm, as listed in Table 2. Therefore, because the 270B alloy in Tables 1 and 2 was grown under the conditions of a 32°C/cm temperature gradient and 270 mm/h growth rate, it could be suggested that this alloy was manufactured at a solidification rate of 14.4°C/min, as listed in Table 2. This value was obtained by the multiple of the temperature gradient and the growth rate. In the same way, the solidification rate in case of the 90B alloy was obtained to be 4.8°C/min.

The lamellar spacing and the solidification rate in both 270B and 90B alloys in Table 2 are showed a linear relationship as presented Fig. 4, and the slope
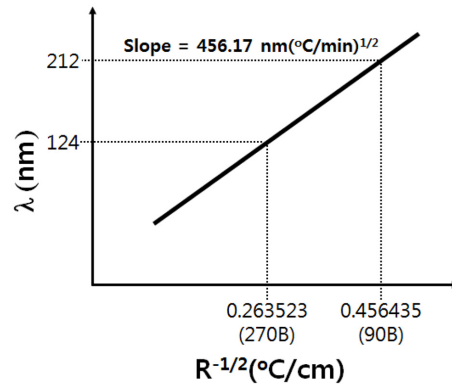
Fig. 4. The linear relationship between the lamellar spacing (λ) and the inverse square root of the solidification rate ($R^{-1/2}$) obtained in the 270B and 90B alloys.

of the line graph calculated in Fig. 4 is about 456.17 nm (^{o}C/min)$^{1/2}$. This slope is supposed to be a constant value in all same alloy composition as previously reported [10-12]. Then, the values of lamellar spacing in the DS alloy produced by using the FZ apparatus were substituted to the linear relationship with the same slope, following the solidification rate in the 90F and 30F alloys could be calculated and results obtained were showed in Fig. 5 and Table 2. The solidification rates of the 90F and 30F alloys were 38.82°C/min and 14.16°C/min, respectively. Because the solidification rates in these
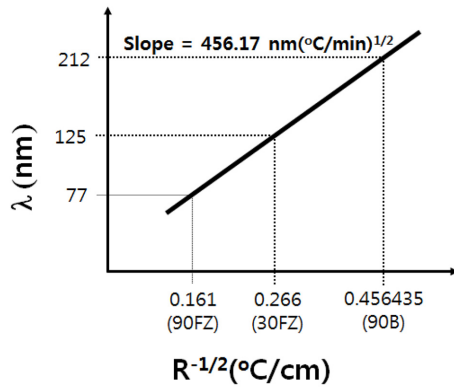
Fig. 5. The linear relationship between the lamellar spacing (λ) and the inverse square root of the solidification rate ($R^{-1/2}$) obtained in the 90F and 30F alloys.

alloys were also obtained by the multiple of the temperature gradient of the FZ apparatus and the growth rate during the DS procedure, the temperature gradient of the FZ furnace could be estimated. As a results, values of 259°C/cm and 283°C/cm in each cases of 90F alloy and 30F alloy were obtained as shown in Table 2, respectively. As we expected, it was found that the temperature gradient of the FZ type DS apparatus was almost 10 times higher than that of the Bridgman type apparatus.

4. Summary

The lamellar microstructures of directionally solidified TiAl alloys were observed by TEM, and the lamellar spacing of the DS alloys was investigated. Then, by using the relationships between the lamellar spacing and the solidification rate, the temperature gradient of the FZ apparatus was estimated. The calculated temperature gradient of the FZ apparatus was about 250~300°C/cm, which were up to ten times higher than that of the Bridgman type apparatus, which was directly measured in this study.

Acknowledgments

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