

(19)



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(11)

EP 2 546 370 A1

(12)

EUROPEAN PATENT APPLICATION

(43) Date of publication:
16.01.2013 Bulletin 2013/03

(51) Int Cl.:
C22C 1/04 (2006.01)
C22C 1/05 (2006.01)
C22F 1/18 (2006.01)

(21) Application number: 12173618.5

(22) Date of filing: 26.06.2012

(84) Designated Contracting States:
AL AT BE BG CH CY CZ DE DK EE ES FI FR GB
GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO
PL PT RO RS SE SI SK SM TR
Designated Extension States:
BA ME

(30) Priority: 13.07.2011 US 201113181732

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(54) Method of making high strength-high stiffness beta titanium alloy

(57) A method of making a high strength, high stiffness beta titanium alloy, comprising introducing boron into a beta titanium alloy to produce TiB precipitates; heat treating the titanium alloy with TiB precipitates by homogenization above the beta transus temperature of the alloy;

subjecting the heat treated alloy to a hot metalworking operation below the beta transus temperature; heat treating the worked alloy with a solution treatment below the beta transus temperature; and ageing the solution treated alloy below the beta transus temperature.

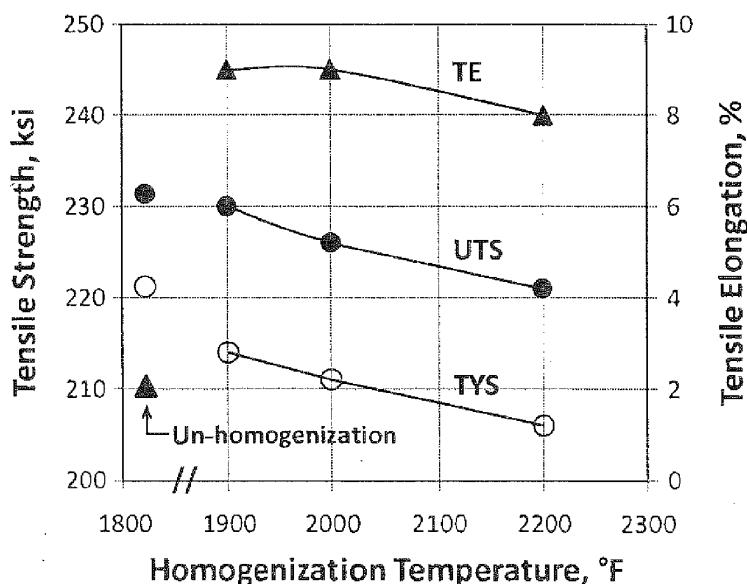


Figure 2

Description**BACKGROUND OF THE INVENTION**5 **Field of the Invention**

[0001] The present invention relates to a method of improving mechanical properties of beta titanium alloys, and more specifically, a method of increasing strength and stiffness of Ti-5Al-5Mo-5V-3Cr (Ti-5553) alloy without debit in ductility.

10 **Description of the Background Art**

[0002] Beta titanium alloys offer improved performance via higher specific strength (strength normalized with density) which enables weight reduction. These alloys find applications in the aerospace industry, e.g., for the structure, landing gear assemblies, and helicopter rotor systems, as described in R.R. Boyer and R.D. Briggs. The Use of Beta Titanium Alloys in the Aerospace Industry, Journal of Materials, Engineering and Performance, Volume 14(6), 2005, pp. 681-685. In these applications, titanium alloys replace steels such as high strength low alloy steel and 4340M steel, providing weight savings along with reduced maintenance due to superior corrosion resistance. The alloy Ti-5Al-5Mo-5V-3Cr (Ti-5553) (all compositions expressed in weight percent) has recently gained an increasing interest as an alternative to the more established alloy Ti-10V-2Fe-3Cr. Ti-5553 alloy offers improved processibility, ability to heat treat in section sizes up to 6 inches and more favorable combination of strength-ductility-toughness. Typical target properties of Ti-5553 in the heat treated condition are ultimate tensile strength of 180 ksi, tensile elongation of 5%, and tensile elastic modulus of 16.2 Msi. Improvements in strength and stiffness of beta titanium alloys would offer improved performance and provide further weight reduction benefit.

[0003] There is a need, therefore, for a new and improved method of increasing the mechanical properties of beta titanium alloys without debits in tensile elongation. The method of present invention meets this need.

BRIEF SUMMARY OF THE INVENTION

[0004] In accordance with the new and improved method of present invention, titanium boride (TiB) precipitates are incorporated into a beta titanium alloy such as Ti-5553, the alloy is then subjected to process steps of homogenization, hot work, and final heat treatment to achieve improvements in mechanical properties compared to the baseline alloy. The boron is introduced into the titanium alloy composition to produce TiB precipitates by a suitable method, such as a pre-alloyed powder metallurgy technique. As an illustrative example, the method of the present invention may be used to increase mechanical properties of Ti-5553 alloy produced via a gas atomized pre-alloyed powder approach.

BRIEF DESCRIPTION OF THE DRAWINGS**[0005]**

40 Figure 1 is a flowchart for making high strength-high stiffness Ti-5553 alloy via a pre-alloyed powder metal approach in accordance with the present invention; and

Figure 2 is a graph of tensile yield strength (TYS), ultimate tensile strength (UTS), and tensile elongation (TE) of enhanced Ti-5553 alloy subjected to different homogenization temperatures and without homogenization. All the samples were final heat treated by solution treating at 1500°F for 1 hour followed by aging at 1100°F for 6 hours.

DETAILED DESCRIPTION OF THE INVENTION

[0006] A new and improved method of increasing mechanical properties of multicomponent beta titanium alloys such as Ti-5553 is described hereinafter.

50 **[0007]** The method described in this disclosure encompasses four critical elements:

1. Incorporation of TiB precipitates into beta titanium alloy matrix;
2. Homogenization heat treatment above the beta transus temperature;
3. Hot work below the beta transus temperature; and
4. Final heat treatment below the beta transus temperature

[0008] Introduction of boron into the titanium alloy composition to produce TiB precipitates can be accomplished by several different methods, such as casting, cast-and-wrought processing, powder metallurgy techniques such as gas atomization and blended elemental approach. Homogenization heat treatment above the beta transus temperature (temperature at which alpha to beta phase transformation is complete) produces equilibrium microstructure that possesses good strength-elongation combination. Conventional hot metalworking operations such as forging, rolling, and extrusion below the beta transus temperature can be used to produce fine-grained microstructure. Final heat treatment comprising solution treatment to precipitate a desired volume fraction of coarse alpha plates followed by ageing to precipitate fine alpha platelets, both conducted below the beta transus temperature, provides the desired strength-elongation combination in the final product. Solution treatment in general is well known to those skilled in the art, as described in "Titanium", G. Lutjering and J.C. Williams, Second Edition, Springer, 2007, page 289.

[0009] The present approach has been practiced by a gas atomization powder metallurgy process flowchart as shown in Figure 1. The boron is added to the molten titanium alloy and the liquid melt is inert gas atomized to obtain titanium alloy powder. Each powder particle contains needle-shaped TiB precipitates distributed uniformly and in random orientations. Titanium alloy powder is consolidated using a conventional technique such as hot isostatic pressing (HIP) at, e.g., 1475°F and 15 ksi for 3 hours to obtain fully dense powder compact. The beta transus temperature of the alloy is determined as 1580°F. The powder compact is homogenized in the temperature range 1900-2200°F to force out super-saturated boron from the titanium lattice and produce equilibrium microstructure. The heat treated compact then is subjected to a metalworking operation such as forging, rolling, or extrusion below the beta transus temperature. A Ti-5553-1B article produced by extrusion of a 3" diameter powder compact into a bar of 0.75" diameter at 1500°F and a ram speed of 120 inch/min is characterized as an example. Extruded bar was heat treated below the beta transus temperature using a combination of solution treatment at 1500°F for 1 hour and gas furnace cooled to room temperature at a cooling rate of about 200°F/minute, plus ageing treatment at 1100°F for 6 hours and air cooled to room temperature.

[0010] By a series of experiments, for a given boron enhancement content, it has been determined that homogenization and ageing are critical steps for achieving improved mechanical property combinations in accordance with the method of the present invention. The influence of homogenization heat treat on room temperature tensile properties of extruded Ti-5553-1B is shown in Figure 2 which shows tensile yield strength (TIS), ultimate tensile strength (UTS) and tensile elongation (TE) of extruded Ti-5553-1B alloy at different homogenization temperatures. The hot work temperature (1500°F), solution treatment (1500°F/1 hour), and ageing (1100°F/6 hours) were kept constant in this study. The alloy without homogenization exhibited high strength (230 ksi ultimate tensile strength) but the tensile elongation was poor (2%). Homogenization in the temperature range 1900-2200°F for 2-4 hours prior to hot work significantly improved the tensile elongation (8% or higher) while maintaining high tensile strength. The tensile strength was higher by up to 50 ksi, or a 28% improvement compared to the typical strength of Ti-5553, as described in "J.C. Fanning", Properties of TIMETAL 555, Journal of Materials Engineering and Performance, Volume 14(6), 2005, pp. 788-791. The tensile modulus of Ti-5553-1B was 19 Msi compared to 16.2 Msi for the baseline Ti-5553, which corresponds to a 17% increase.

[0011] The influence of ageing treatment on room temperature tensile properties of extruded Ti-5553-1B for different homogenization temperatures is demonstrated in Table 1 hereinafter. The hot work temperature (1500°F), solution treatment (1500°F/1 hour), and ageing time (6 hours) were kept constant in this study. Upon ageing, tensile strength increased by 50-60 ksi, tensile modulus increased by 4-5 Msi without debit in tensile elongation compared to the no post heat treat condition. By a suitable choice of homogenization temperature and ageing temperature, optimum strength-modulus-ductility combinations can be achieved as shown in Table 1.

Table 1: Tensile properties of Ti-5553-1B alloy homogenized at different temperatures

and tested without and with final heat treatment of solution treatment plus ageing.

Solution treatment of 1500°F for 1 hour was used. (TYS: Tensile Yield Strength, UTS:
 5 Ultimate Tensile Strength, TE: Tensile Elongation, RA: Reduction of Area, TM: Tensile
 Modulus).

T_{Homogenization}	T_{Ageing}	TYS	UTS	TE	RA	TM
°F	°F	ksi	ksi	%	%	Msi
1900	None	160	169	9	18	14.5
	1100	214	230	9	13	19.0
2000	None	152	159	9	25	14
	1100	211	226	9	16	19.1
2200	None	151	158	5	27	13.3
	1100	206	221	8	12	18.7

[0012] While the invention has been described in connection with what is presently considered to be the most practical and preferred embodiments, it is to be understood that the invention is not to be limited to the disclosed embodiments, 25 but on the contrary, is intended to cover various modifications and equivalent arrangements included within the spirit and scope of the appended claims.

Claims

1. A method of making a high strength, high stiffness beta titanium alloy, comprising:
 introducing boron into a beta titanium alloy to produce TiB precipitates;
 heat treating the titanium alloy with TiB precipitates by homogenization above the beta transus temperature of
 35 the alloy;
 subjecting the heat treated alloy to a hot metalworking operation below the beta transus temperature;
 heat treating the worked alloy with a solution treatment below the beta transus temperature; and
 ageing the solution treated alloy below the beta transus temperature.
2. The method of Claim 1 wherein the TiB precipitates are produced in the alloy by casting, cast-and-wrought processing, powder metallurgy techniques, e.g., gas atomization, or blended elemental approach.
3. The method of Claim 2 wherein the boron is added to a molten titanium alloy and the liquid melt is atomized to obtain titanium alloy powder containing TiB precipitates, and the titanium alloy powder is consolidated to obtain a fully
 45 dense powder compact.
4. The method of Claim 3 wherein the titanium alloy powder is consolidated by hot isostatic pressing.
5. The method of Claim 1 wherein the beta transus temperature of the alloy is about 1580°F and the alloy is heat treated by homogenization at a temperature range of about 1900-2200°F for 2-4 hours.
6. The method of Claim 5 wherein the hot metalworking is forging, rolling or extrusion at a temperature of about 1500°F.
7. The method of Claim 6 wherein the heat treated alloy is extruded at a ram speed of approximately 120 inch/min.
8. The method of Claim 7 wherein the heat treated alloy is extruded from a powder compact into a bar.
9. The method of Claim 5 wherein the worked alloy is heat treated with a solution treatment at approximately 1500°F

for about 1 hour and cooled to room temperature.

10. The method of Claim 9 wherein the heat treated and worked alloy is gas furnace cooled to room temperature at a cooling rate of about 200°F/minute.

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11. The method of Claim 9 wherein the solution treated alloy is aged at about 1100°F for about 6 hours.

12. The method of Claim 11 wherein the aged alloy is air cooled to room temperature.

10 13. The method of Claim 1 wherein the heat treating of the titanium alloy by homogenization improves the tensile elongation while maintaining the tensile strength of the titanium alloy.

14. The method of Claim 1 wherein the ageing of the solution treated alloy increases the tensile strength and tensile modulus of the alloy without a reduction in tensile elongation.

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15. The method of Claim 1 wherein the titanium alloy is Ti-5553.

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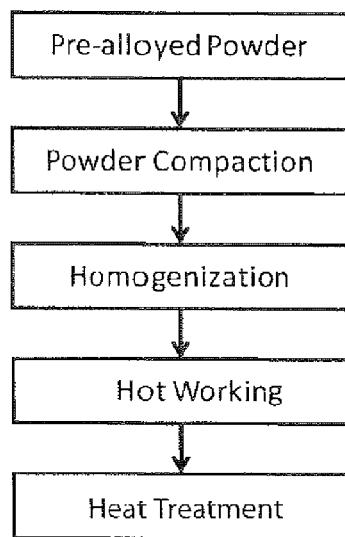


Figure 1

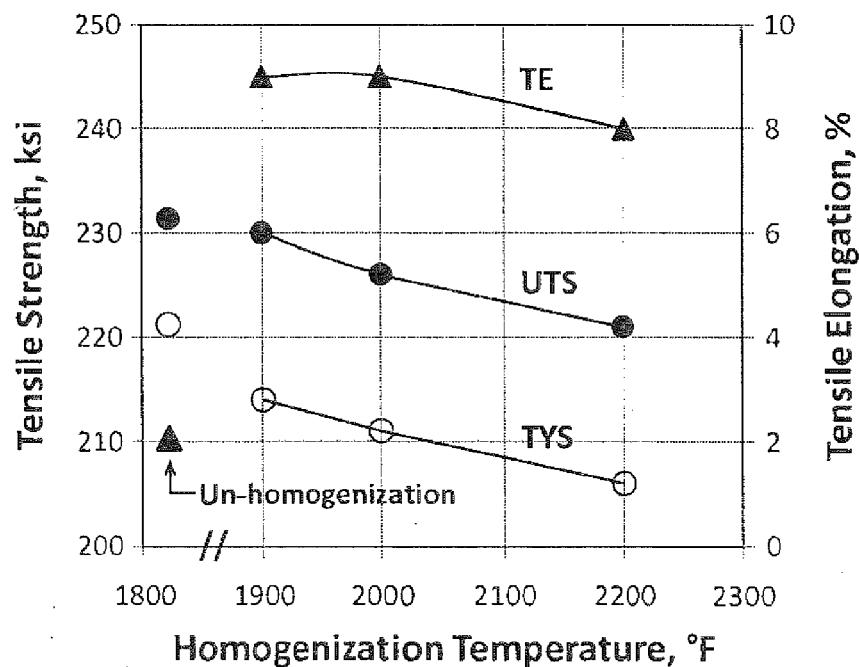


Figure 2



EUROPEAN SEARCH REPORT

Application Number
EP 12 17 3618

DOCUMENTS CONSIDERED TO BE RELEVANT			CLASSIFICATION OF THE APPLICATION (IPC)
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	
X	US 2010/180991 A1 (HERITIER PHILIPPE [FR] ET AL) 22 July 2010 (2010-07-22) * paragraphs [0001], [0003], [0004], [0007] - [0009], [0023] - [0030], [0032] * ----- X WO 2007/142837 A1 (FMW COMPOSITE SYSTEMS INC [US]; MIRACLE DANIEL B [US]; TAMIRISAKANDALA) 13 December 2007 (2007-12-13) * paragraphs [0022], [0023], [0027]; figure 3(b) * ----- A WO 2005/060631 A2 (UNIV OHIO [US]; MIRACLE DR DANIEL B [US]; TAMIRISAKANDALA DR SESHACHAR) 7 July 2005 (2005-07-07) * the whole document * -----	1-5,8-15 1-5,8, 13,15 1-15	INV. C22C1/04 C22C1/05 C22F1/18
			TECHNICAL FIELDS SEARCHED (IPC)
			C22C C22F
The present search report has been drawn up for all claims			
2	Place of search Munich	Date of completion of the search 30 November 2012	Examiner Brown, Andrew
CATEGORY OF CITED DOCUMENTS <p> X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document </p> <p> T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document </p>			

**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

EP 12 17 3618

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on. The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

30-11-2012

Patent document cited in search report		Publication date		Patent family member(s)		Publication date
US 2010180991	A1	22-07-2010		CA 2748380 A1		01-07-2010
				CN 102317484 A		11-01-2012
				EP 2379760 A1		26-10-2011
				FR 2940319 A1		25-06-2010
				JP 2012514129 A		21-06-2012
				US 2010180991 A1		22-07-2010
				WO 2010072972 A1		01-07-2010
<hr/>						
WO 2007142837	A1	13-12-2007		CN 101501228 A		05-08-2009
				EP 2038443 A1		25-03-2009
				KR 20090029782 A		23-03-2009
				US 2007286761 A1		13-12-2007
				WO 2007142837 A1		13-12-2007
<hr/>						
WO 2005060631	A2	07-07-2005		CN 101080504 A		28-11-2007
				EP 1697550 A2		06-09-2006
				JP 4890262 B2		07-03-2012
				JP 2007519822 A		19-07-2007
				KR 20120064687 A		19-06-2012
				US 2011146853 A1		23-06-2011
				US 2012180913 A1		19-07-2012
				WO 2005060631 A2		07-07-2005
<hr/>						

REFERENCES CITED IN THE DESCRIPTION

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Non-patent literature cited in the description

- **R.R. BOYER ; R.D. BRIGGS.** The Use of Beta Titanium Alloys in the Aerospace Industry. *Journal of Materials, Engineering and Performance*, 2005, vol. 14 (6), 681-685 **[0002]**
- **G. LUTJERING ; J.C. WILLIAMS.** Titanium. Springer, 2007, 289 **[0008]**
- **J.C. FANNING.** Properties of TIMETAL 555, *Journal of Materials Engineering and Performance*, 2005, vol. 14 (6), 788-791 **[0010]**