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Grain refinement mechanism in advanced γ-TiAl boron-alloyed structural intermetallics: The direct observation

A.V. Kartavykh [a,b,*], M.V. Gorshenkov [a], D.A. Podgorny [a]

[a] National University of Science and Technology “MISIS”, Leninsky pr. 4, 119049 Moscow, Russia
[b] Technological Institute for Superhard and Novel Carbon Materials (TISNCM), 7a Centralnaya str., 142190 Troitsk, Moscow, Russia

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ABSTRACT

The synthesis of Ti–44Al–5Nb–2Cr–1.5Zr–0.4B–0.07La and Ti–44Al–5Nb–1Cr–1.5Zr–1B–0.17La (at%) intermetallics was performed from pure metals by the electron beam semi-continuous casting technique. Alloys possess the convoluted microstructure whose refinement is progressive with the boron content increase. The specimens were analyzed using SEM, EBSD and EDX techniques. The boron alloying causes precipitation of micro-dimensional complex borides within solidifying melt. These particles-inoculants promote the formation of finer microstructure. *Auger* spectrometry was applied for quantitative elemental analysis of borides. It was proven that the origin of structure refinement consists in solid-phase germination of α_2-Ti_3Al-phase on (Ti,Nb)B ribbon-like colonies with subsequent growth of α_2-laths through γ-TiAl matrix. We fixed several consecutive stages of microstructure refinement process. The description of these stages is supported with the crystallographic and compositional characterization of microstructural constituents. The work provides new data for microstructure/mechanical properties engineering of studied materials.

1. Introduction

Lightweight multicomponent γ-TiAl-intermetallics are creep resistant to high temperatures (~1100 K), so being advanced materials for turbine blades of aircraft engines and gas-burning power-generation plants [1]. Nb alloying significantly improves high temperature properties of TiAl based alloys, whereas minor additions of Cr and Zr enhance their oxidation resistance within turbine gas flow. Lanthanum addition was applied firstly by us for the internal gettering of undesired oxygen impurity [2]. Generally, finer microstructure provides better performance of an alloy that requires the specific two-phase γ-TiAl+α_2-Ti_3Al polycrystalline pattern creation in cast items. That is why the attention of researchers is paid to microstructure improvement by modifying doping of alloy with boron. The precise boron alloying causes precipitation of micro-dimensional Ti-based borides within solidifying melt. These particles-inoculants act as seed centers of crystallization (and/or phase transformation), promoting the formation of finer microstructure in an alloy when casting/cooling. Despite the practical application in cast TiAl, no clear-cut understanding of the grain refinement mechanism associated to boron additions is available today; this process has never been observed directly.

As it was experimentally observed in [3], in Ti–45Al–*x*B alloys (where $x = 0.3$–0.8 at%) the boride precipitated from the melt at the temperature above liquidus, i.e. before the appearance of primary β (Ti)-phase. According to the results from the solidified Nb-alloying derivative Ti–44Al–8Nb–1B, boride starts to precipitate from the liquid either simultaneously with, or closely following the β-phase [4]. That is why two basic hypotheses remain usable up to date. Cheng [5] explain grain refinement by primary nucleation of the β (Ti) phase on borides, that is run rather well in heavily alloyed (1 at% of boron and higher) pro-peritectic alloys. Controversial hypothesis stated that grain refinement is not based on refining the β(Ti) phase, but rather the α(Ti) phase, especially in the alloys of peritectic composition. The role of boride in grain refinement of such alloys is to inoculate α when the β phase decomposes [4,6,7]. As considered in [6], grain refinement occurred during β-to-α transformation through boride assisted solid-phase α(Ti) nucleation rather than during solidification.

Present paper reports the detailed characterization and analysis of microstructurally improved new alloys, with the particular study of boride inoculants appeared within the alloy matrix, along with the direct observation of structure refining mechanism.

* Corresponding author at: National University of Science and Technology “MISIS”, Leninsky pr. 4, 119049 Moscow, Russia. Tel.: +7 495 5126091; fax: +7 499 4006260.
E-mail address: karta@korolev-net.ru (A.V. Kartavykh).

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2. Materials and methods

The synthesis of alloys with Ti–44Al–5Nb–2Cr–1.5Zr–0.4B–0.07La and Ti–44Al–5Nb–1Cr–1.5Zr–1B–0.17La (at%) nominal compositions has been performed by the electron beam semi-continuous casting technique in vacuum using the sticks of pure initial metals. Lanthanum hexaboride LaB_6 was used as the boron-contained additive. The alloys were produced as billets of 160 mm in diameter and a mass of 15 kg. The final stage of casting represents upward vertical directional melt solidification in copper “cold” crucible. That allows the controlling both the billet’s withdrawal rate, and related cooling rate within the same process. Thus, cooling rate was varied in the range from 1.3 to 3 K s^{-1} along an ingot in the course of solidification. More detailed description and schematic of applied cast technique could be found in [2].

The alloys were analyzed using the *JEOL JSM*6610 electron microscope, equipped with *AZtecSynergy* EBSD and EDX systems. The 1-μm diamond polishing of slices was done using *Buehler* equipment and consumables. No chemical etching was applied for the overall microstructure pattern reviewing by SEM. The deep

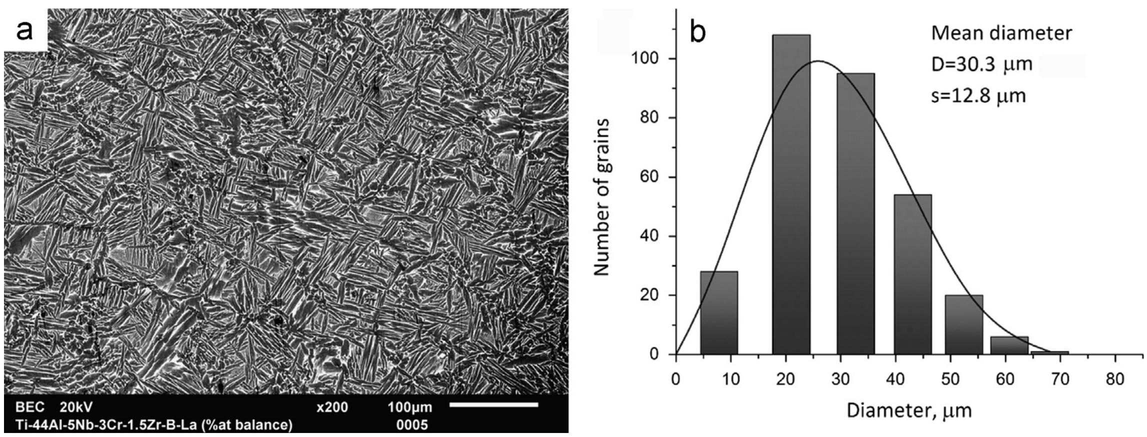

Fig. 1. (a) SEM micrograph of Ti–44Al–5Nb–1Cr–1.5Zr–1B–0.17La (at%) alloy; (b) the histogram of grains distribution by the diameter obtained by the processing of this image, and its approximation by the normal statistical distribution curve.

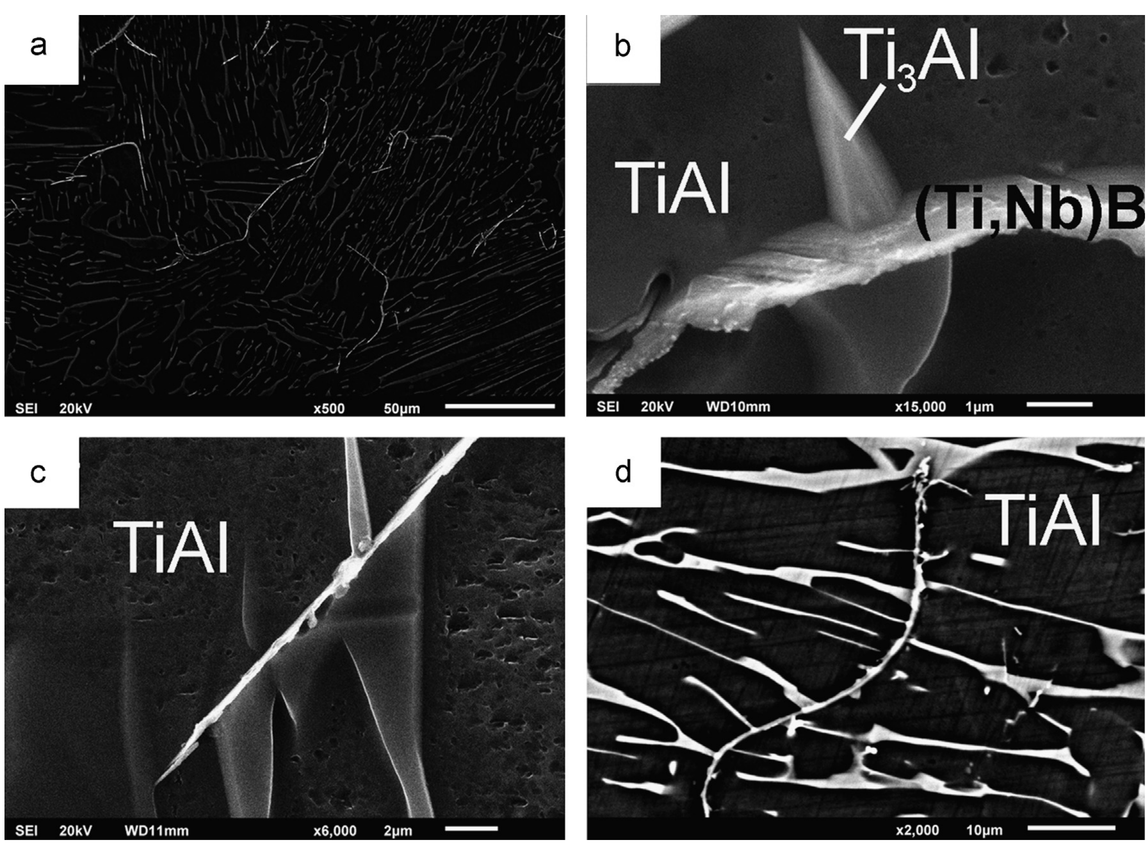

Fig. 2. Boride ribbons resolved in Ti–44Al–5Nb–2Cr–1.5Zr–0.4B–0.07La by SEM (a) and three consecutive stages of structure refining mechanism development: the event of solid-state germination (seeding) of α_2-Ti_3Al phase on boride facet (b); morphological development of α_2-laths and their growth through γ-TiAl matrix (c); formation of new grain boundary by elongated curved boride surrounded by fine $\gamma+\alpha_2$ phase structure inside the delimited grains (d). The micrographs (b), (c) and (d) derived from the billet parts which being cooled at the rates 3.0, 2.2 and 1.3 K s^{-1}, respectively.