Intermetallics 102 (2018) 120–131

Contents lists available at ScienceDirect

Intermetallics

journal homepage: www.elsevier.com/locate/intermet

Lattice parameter evolution during heating of Ti-45Al-7.5Nb-0.25/0.5C alloys under atmospheric and high pressures

Xi Li[a,b], Rian Dippenaar[a], Ayumi Shiro[c], Takahisa Shobu[d], Yuji Higo[e], Mark Reid[a,b], Hiroshi Suzuki[f], Koichi Akita[f], Ken-Ichi Funakoshi[g], Klaus-Dieter Liss[a,b,f,h,i,*]

[a] School of Mechanical, Materials, Mechatronic and Biomedical Engineering, University of Wollongong, NSW, 2522, Australia
[b] Australian Nuclear Science and Technology Organisation, Lucas Heights, NSW, 2234, Australia
[c] Quantum Beam Science Research Directorate, National Institute for Quantum and Radiological Science and Technology, Kouto, Sayo, Hyogo, 679-5148, Japan
[d] Materials Sciences Research Center, Japan Atomic Energy Agency, Kouto, Sayo, Hyogo, 679-5148, Japan
[e] SPring-8, Japan Synchrotron Radiation Research Institute, Kouto, Sayo, Hyogo, 679-5198, Japan
[f] Materials Sciences Research Center, Japan Atomic Energy Agency, Tokai, Ibaraki, 319-1195, Japan
[g] Neutron Science and Technology Center, Comprehensive Research Organization for Science and Society (CROSS), Tokai, Ibaraki, 319-1106, Japan
[h] Guangdong Technion – Israel Institute of Technology, Shantou, 515063, China
[i] Technion – Israel Institute of Technology, Haifa, 32000, Israel

ARTICLE INFO

Keywords:
Synchrotron radiation
TiAl
High pressure
High temperature
Lattice strain

ABSTRACT

Lattice strain evolution during deformation processing of Ti-Al alloys at high temperature is important in terms of its microstructural evolution and microstructural stability. It is shown here that careful evaluation of lattice parameters is critical for the understanding of thermal expansion, crystallographic order, chemical composition and response to pressure, allowing to identify phase transitions and segregation, in addition to the measurement of the more conventional quantities phase composition and order parameter. The lattice parameters of Ti-45Al-7.5Nb-0.25/0.5C (at. %) alloys were calculated using both Rietveld and single peak fitting methods from data obtained by *in-situ* synchrotron diffraction experiments at high temperature under atmospheric and high pressure respectively. The lattice strain evolution as a function of temperature in a Ti-45Al-7.5Nb-0.25C (at. %) alloy under high pressure was compared with that of a Ti-45Al-7.5Nb-0.5C (at. %) alloy heated under atmospheric pressure. The contribution of each of the four lattice strain factors is semi-quantitatively assessed in the temperature range investigated.

1. Introduction

Titanium aluminides are attractive candidate materials for applications in the automotive industry, and more importantly for applications at high temperature in aerospace industries, mainly due to their low density and excellent mechanical properties [1–3]. A new route for the processing of titanium aluminide components under high pressure has been proposed [4], for example, by utilizing a 0.8 GN forging press to manufacture large aerospace products [5] or a new 0.54 GN die-forging press currently being commissioned [6]. Since these forming processes operate at elevated temperature and pressure, it is imperative that the microstructural integrity of the work-pieces be maintained, especially by limiting grain growth during high-temperature processing and minimizing the development of internal stresses during forging operations.

Processing in the ($\alpha_2/\alpha + \gamma$) phase-field remains a challenge due to crystalline anisotropy and the presence of intermetallic bonds. Appel et al. [7] pointed out that: "The important difference with alloys that solidify completely through β and those that subsequently precipitate α on further cooling, is that the α that forms from the β can do so with up to 12 different orientation variants". Thus, a large β grain can be divided into lamellar colonies with up to 12 different orientations. This effect, termed "crystal partitioning" [8,9], can therefore lead to significant grain refinement of castings and significantly reduced texture. The β-phase is highly isotropic and independent slip systems can operate during dynamic recovery as experimentally confirmed by Liss et al. [10] in an *in-situ* synchrotron X-ray study. The β-solidifying alloys being studied in the present program have a fine and homogenous microstructure and are therefore easier to forge than conventional ($\alpha_2/\alpha + \gamma$) alloys [7,11,12]. It is possible to stabilize the β-phase by the addition of selected alloying elements, but it has recently been established that the β-phase can also be physically induced by the

* Corresponding author. Guangdong Technion - Israel Institute of Technology, 241 Daxue Lu, Jinping Qu, Shantou 515063, Guangdong, China
E-mail addresses: kdl@gtiit.edu.cn, kdliss@technion.ac.il, liss@kdliss.de (K.-D. Liss).

https://doi.org/10.1016/j.intermet.2018.08.011
Received 1 May 2018; Received in revised form 21 August 2018; Accepted 23 August 2018

application of high hydrostatic pressure at high temperature. However, the question as to how this newly formed β-phase would affect the lattice strains in the α_2/α and γ phases has not been answered [13] and it is important to answer this question because the lattice strains have a determining influence on the mechanical properties of the product. Moreover, the evaluation of lattice parameters is a very sensitive measurement to determine phase transformations of various kinds [14–16] and can reveal segregation pathways in phase diagrams [17]. Therefore, under the aspect of lattice parameter evolution as the main complementary evaluation we re-visit data from two *in-situ* heating experiments, to compare (i) a high-energy synchrotron radiation study on Ti-45Al-7.5Nb-0.5C under atmospheric pressure [21] with (ii) an energy-dispersive synchrotron X-ray diffraction experiment on Ti-45Al-7.5Nb-0.25C under high pressure at 9.6 GPa [13].

Fig. 1(a) shows a section through the Ti-Al binary phase diagram [18], while a section through a Ti-Al-7.5 at. % Nb alloy, proposed by Chladil et al. [19] is shown in Fig. 1(b). The alloy used in the present study is schematically shown by the vertical line at 45 at. % Al. It is important to note that this section through the phase diagram applies to atmospheric pressure and to the knowledge of the authors, the extent to which pressure changes the pertaining phase equilibria has not been determined as yet.

Since we study a Ti-Al-Nb-C alloy under conditions of severe-plastic deformation and high temperature, it is important to identify the most critical parameters that determine microstructural evolution and stability. One important variable is lattice parameter evolution, since for example, a change in the *c/a* ratio of the α-lattice has a determining influence on the pertaining slip and twinning deformation mechanisms. Moreover, changes in the lattice parameter impact on orientation relationships and have an influence on interphase stress development. In addition, the mechanism and morphology of phase transformations need to be taken into account since they play an important role in achieving microstructural stability.

Although the lattice parameter evolution plays a pivotal role in assessing the high-temperature behavior of titanium-aluminides, as argued above, only little information has been traced to date, with the notable exception of the *in-situ* studies early by Shull et al. [20], then by Yeoh et al. [21] and more recently that of Liss et al. [13].

The very early work of Shull et al. in 1990 reports on the first *in-situ* investigation of titanium aluminides at high temperature, focusing on the experimental determination of phase fields, while lattice parameter evolution is traced. In 2007, Yeoh et al. [21] reported the changes occurring in the *c/a* ratio of the lattice parameters in a Ti-45Al-7.5Nb-0.5C alloy during heating at atmospheric pressure. She suggested that as far as X-ray analyses are concerned, a simplification should be made by assuming that the α_2- (ordered) and the α-phase (disordered) be regarded as a single phase since X-rays cannot clearly distinguish between an ordered and disordered structure.

Liss et al. [13] recently conducted an *in-situ* X-ray diffraction experiment on a Ti-45Al-7.5Nb-0.25C alloy under high hydrostatic pressure. They studied the phase evolution of a Ti-45Al-7.5Nb-0.25C alloy as a function of time under high pressure and high temperature within a synchrotron X-ray source (SPring-8 beamline BL04B1, run number M1472). The *in-situ* diffactograms are displayed in Fig. 2 which have been analyzed by the Rietveld method using MAUD (*Material Analysis Using Diffraction software* [22,23]) for the evolution of phase fractions as a function of temperature, shown in Fig. 3. Also shown are the phase fractions determined by Yeoh et al. [21] in a roughly similar alloy Ti-45Al-7.5Nb-0.5C, but under standard atmospheric conditions. The two alloys have been manufactured under identical conditions and the two *in-situ* experiments were conducted under normal atmospheric and high pressure respectively. However, it is important to note that carbon can have a significant influence on phase evolution in these alloys and for this reason care need to be taken in comparing the alloys containing 0.25 at. % C and 0.5 at. % C respectively. For example, the eutectoid temperature, T_{eu}, is increased by 20 K from 1453 K to 1473 K, but the respective γ-solvus, $T_{\gamma,solv}$, 1565 K and 1566 K respectively [24] remains essentially constant. Notwithstanding these differences, the two alloys can be compared with respect to their respective pressure-induced behaviors. The *fcc*-based, ordered γ-phase of $L1_0$ structure, co-exists with an *hcp*-based, ordered α_2-phase of $D0_{19}$ structure at room temperature. Upon heating, the α_2-phase undergoes an inverse eutectoid order-disorder transition to form a fully disordered hexagonal α-phase at T_{eu}. The fraction of the γ-phase decreases upon heating and finally transforms fully into the disordered α-phase at $T_{\gamma,solv}$. Salient features when the Ti-45Al-7.5Nb-0.25C alloy is heated under a pressure of 9.6 GPa, are the appearance of the *bcc* β-phase in an $(\alpha + \alpha_2 + \beta + \gamma)$ field and the dissolution of γ phase at $T_{\gamma,solv}$ to form $(\alpha + \beta)$.

Liss et al. [13] analyzed the lattice strain development in the Ti-45Al-7.5Nb-0.25C alloy at 310 K at three pressures, up to 9.6 GPa (series numbers 2, 4 and 7 in Fig. 2). They calculated the changes in lattice parameter of the γ- and α_2/α-phases as a function of pressure at this temperature [13] and argued that at room temperature, the volume response to pressure is accommodated by the phase transformation $\gamma \rightarrow \alpha_2$, rather than by volumetric strain. They further determined some crystallographic aspects, specifically lattice strain and atomic order, at room temperature, but did not determine lattice strain evolution during heating at high pressure, which is subject of the current project. It is the dearth of information of this parameter, critical to processing at high temperature and pressure, which prompted the present investigation.

The overarching aim of the present work was the re-visit of the experimental data of both experiments [13,27] in order to compare the behavior of the selected alloys under atmospheric and high pressure respectively.

The specific aims were:

- to determine the lattice parameter evolution as a function of

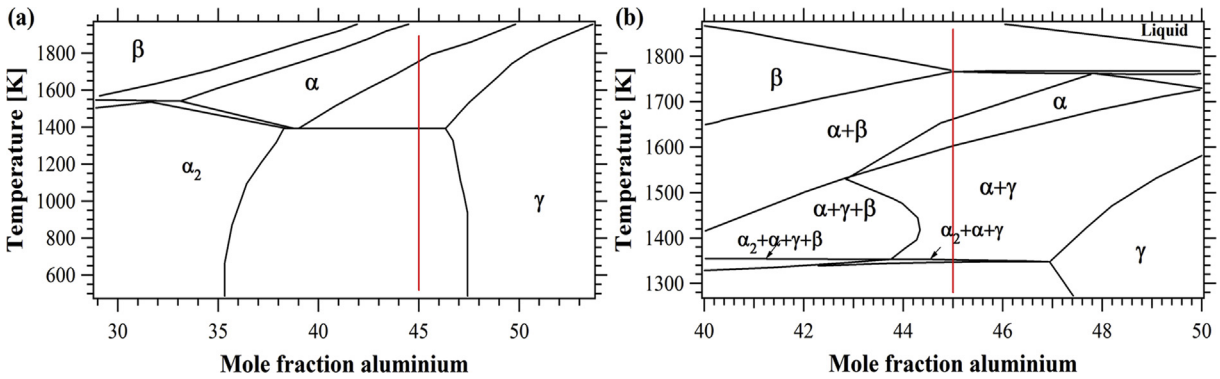

Fig. 1. (a) Binary phase diagram of Ti-Al [18]; (b) Section through a proposed phase diagram of the Ti-Al-Nb alloy system for an alloy containing 7.5 at. % Nb [19].