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Carbon distribution in multi-phase γ-TiAl based alloys and its influence on mechanical properties and phase formation

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Abstract—Advanced intermetallic γ-TiAl based alloys are attractive light-weight materials for high-temperature application. In order to extend their service temperature limits, alloying with low-density elements, such as C, is of particular interest and has been shown to effectively increase high-temperature strength as well as creep resistance.

In the present study the local chemical composition of the constituent phases of the so-called TNM alloy and a C-containing derivative thereof is characterized by atom probe tomography. In both alloys Mo is found to preferentially locate in the β_o phase, in contrast to Nb, which is dispersed in similar levels in all phases. In the C-containing alloy, C is enriched in the α_2 phase, dissolved in the γ phase, but depleted in the β_o phase. Furthermore, the investigation of interfaces through site-specific sample preparation reveals segregation of C at phase interfaces and their close vicinity. Finally, a correlation of the mechanical properties with the C distribution is established by nanoindentation technique. Both the γ and the α_2 phase significantly harden through the addition of C, which is in good agreement with the C concentration present within these phases as observed by atom probe tomography. However, the β_o phase softens through the addition of C, which is not a direct consequence of the C distribution, but follows from the absence of finely dispersed ω_o particles in the β_o phase of the C-containing alloy.

Keywords: Titanium aluminides; Atom probe tomography (APT); Interfacial segregation; Nanoindentation; Omega phase

1. Introduction

Advanced intermetallic γ-TiAl based alloys have found application as structural high-temperature materials for turbine blades in aero engines and turbocharger wheels in automotive combustion engines. Among the exceptional properties of this alloy class that especially facilitate their applicability are their low density of about 3.8–4.1 g cm^{-3}, their high creep and oxidation resistance as well as their high strength and modulus retention at elevated temperatures [1–3]. The service temperature limit of current engineering TiAl alloys is around 750 °C, mostly due to insufficient creep resistance at temperatures beyond [3]. However, a significant enhancement of high-temperature strength and creep properties can be achieved by alloying with light elements, which has been extensively investigated by numerous researchers [4–18]. Especially C and Si additions improve the mechanical properties, particularly at elevated temperatures. The main focus of the present study is the effect of C as an alloying element on chemical composition and local hardness of the constituent phases. Kawabata et al. [4], Tian and Nemoto [5] and Appel et al. [1,8,9] reported on the positive effect of C on the tensile properties of intermetallic TiAl alloys. They observed a substantial increase in strength of C doped alloys due to solid solution hardening and precipitation of fine carbides. Worth et al. [6] comprehensively investigated the effect of C addition on the creep resistance and observed a significant reduction of the minimum creep rate in C-containing alloys exhibiting different microstructures. In their study solid solution hardening was identified as the active mechanism. More recently, several studies focused on the properties of C-containing advanced γ-TiAl based alloys, whereby effects pertaining to workability [13], microstructural evolution [16] and carbide precipitation [15,18] were addressed. Through the addition of C the improvement of mechanical properties is either achieved by solid solution hardening or by precipitation hardening. The predominant mechanism is determined by the exact chemical composition and the heat-treatment applied, which results in the necessity of a well-considered adjustment of processing and heat-treatment parameters. In particular in the case of binary TiAl alloys the solubility limits for interstitial elements are small [19,20]. Menand et al. [21] and Lefebvre

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et al. [22] postulated that the interstitial solubility limit of the γ phase is strongly influenced by the derivation from the stoichiometric composition. They argued that a decreasing aluminum content, compared to the stoichiometric composition, results in the creation of Ti antisite defects which in turn causes the introduction of Ti_6-octahedral cavities. This change in local chemical environment is favorable for the incorporation of interstitial atoms such as C, N or O and, therefore, the solubility limit of the γ phase increases toward the aluminum-lean off-stoichiometric composition. Furthermore, the introduction of ternary elements potentially increases the solubility limit for interstitials in the γ phase, mainly depending on the lattice site preference. For example, Scheu et al. [14] addressed the C solubility of the γ phase of a Nb- and C-containing γ-TiAl based alloy experimentally by atom probe tomography (APT) and compared the obtained results with theoretical estimates. They concluded that Nb effectively increases the interstitial solubility limit of the γ phase due to its preferred location on Ti sites of the $L1_0$-structure (see also e.g. [23]) and, hence, an increased number of Ti_6-octahedral cavities.

In the present study we aim at the determination of the alloying element distribution, including the C distribution, within all constituent phases of advanced multi-phase γ-TiAl based alloys by APT. Moreover, site-specific sample preparation of APT tips containing phase interfaces is utilized in order to characterize and quantify segregational effects. Concomitant microstructural investigations are conducted using transmission electron microscopy (TEM) and transmission electron backscatter diffraction (t-EBSD) in order to illuminate effects with regard to phase formation.

The influence of the C distribution on the mechanical properties of the individual phases in TiAl alloys was studied by means of nanoindentation technique [24]. The obtained results are utilized for a correlation between the C distribution and the hardness of a C-containing multi-phase γ-TiAl based alloy.

2. Materials and methods

2.1. Alloy compositions and processing

Two materials based on the TNM alloying concept [25] were investigated within this study. The chemical composition of the investigated alloys is summarized in Table 1. Throughout this publication all chemical compositions are given in atomic percent (at.%), unless indicated otherwise.

The alloys were produced by GfE Metalle und Materialen GmbH, Germany. Details about the production route can be found in Refs. [3,26]. In the production process the ingot of the TNM alloy was remelted twice in a vacuum arc furnace, whereas the ingot of the TNM0.75C alloy was remelted once. Thereafter the ingots were melted again using vacuum arc remelting (VAR) in the case of the TNM alloy and vacuum induction melting (VIM) in the case of the TNM0.75C alloy. Afterward the TNM alloy was cast using centrifugal casting, whereas the TNM0.75C alloy was cast using gravity casting. Subsequently, the ingots were subjected to a hot isostatic pressing (HIP) procedure at 1200 °C for 4 h at a pressure of 200 MPa and finally cooled to room temperature by furnace cooling. Then the ingots were conventionally hot-forged at Böhler Schmiedetechnik GmbH & Co KG, Austria, by the so-called hot-die forging process [27,28] and subsequently subjected to a homogenization heat-treatment around the eutectoid temperature for several hours followed by air cooling to achieve a structurally homogeneous microstructure [29]. The heat-treatments were conducted under atmospheric conditions in a high-temperature furnace Carbolite RHF 1600.

Table 1. Chemical composition of the investigated γ-TiAl based alloys in at.%.

	Ti	Al	Nb	Mo	B	C
TNM	bal.	43.4	4.3	1.2	0.1	0.02[a]
TNM0.75C	bal.	43.7	4.1	1.1	0.1	0.78

[a] Unintentional impurity in the TNM alloy from production process.

2.2. Experimental techniques

Microstructural images were taken using a field emission gun (FEG) dual focused ion beam (FIB) device Versa 3D DualBeam™ from FEI in back-scattered electron (BSE) mode, on electrolytically polished samples (A3-electrolyte by Struers). All images shown in this paper were taken using a 20 kV acceleration voltage.

The FIB workstation was also used for microstructural characterization using t-EBSD conducted in the vicinity of APT tip apexes. This method has been thoroughly assessed in Ref. [30]. For t-EBSD investigations the FIB workstation is equipped with an EDAX Hikari XP EBSD System. The EDAX OIM Data Collection 7 software was utilized in combination with the EDAX OIM Analysis 7 software for the subsequent evaluation of the data files.

Furthermore, the FIB workstation, equipped with a platinum gas injection system and a micromanipulator (Omniprobe™ 100 by Oxford Instruments), was employed for the preparation of tip-shaped APT specimens. Two different approaches were selected to prepare tips suitable for APT. Firstly, the lift-out technique [31,32] was used for site-specific preparation of individual phases with a low volume fraction and of selected interfaces of interest. Secondly, FIB milling of previously electrolytically etched pre-tips was used in combination with t-EBSD in order to place a specific microstructural constituent in the vicinity of the APT tip's apex. By the lift-out technique wedges were prepared of the region of interest using an acceleration voltage of 30 kV and a suitable current for a balance between cutting time and re-deposition. Thereafter, these wedges were removed with the micromanipulator. Subsequently, the wedges were transferred to pre-shaped posts and fixed by a Pt weld. Prior to this procedure the material was protected from Ga implantation by Pt deposition layer. In order to shape the specimens for the examination in the APT, annular milling was performed using an acceleration voltage of 30 kV to obtain a defined tip radius and shank angle (<100 nm in diameter and 10° of shank angle). Cleaning steps using lower acceleration voltages, 5 kV and 2 kV, were applied in order to minimize Ga implantation and amorphization of the tip's surface.