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Variant selection during nucleation and growth of γ-massive phase in TiAl-based intermetallic alloys

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Abstract

This work deals with the mechanisms of nucleation and growth of γ-massive phase in TiAl-based intermetallic alloys. In particular, it focuses on the process of variant selection operating at both stages of the transformation. Small γ-massive domains produced by rapid cooling are extensively characterized by high-resolution electron backscatter diffraction. This large data set allows a statistical analysis of nucleation sites, according to different crystallographic configurations. It is established that, whatever the nucleation sites, i.e., grain boundaries or triple junctions, a coherent facet is always found presenting a Blackburn orientation relationship (BOR) between the γ nucleus and the α parent grain. Moreover, some γ nuclei can additionally present another semicoherent facet with an approximate BOR with the α host grain. A new nucleation mechanism, called "co-nucleation", is highlighted for this type of double-faceted nucleus. Variant selection during nucleation is discussed for both types of nuclei, in terms of minimization of interface energy. In addition, it is shown that growth of γ-massive domains from their initial nucleus always involves successive {1 1 1} twinning. Variant selection also occurs either at the nucleus growth stage or during the development of successive twin generations, and is discussed in terms of interphase boundary mobility.

Keywords: Massive transformation; Variant Selection; Crystallography; Nucleation

1. Introduction

Massive transformation, as defined by Massalski [1], is a "composition invariant, interface-controlled diffusional phase transformation, involving a characteristic irregular patchy microstructure and frequent faceting and ledges, but not necessarily involving lattice orientation relationships". This type of transformation was first observed by Phillips [2], and its main characteristics were initially established by Massalski in a range of Cu alloys [3]. Since then, massive-like transformations have been reported in a number of alloys [3–28,31–34], with major contributions from Massalski and co-workers [1,3–8] and Aaronson and co-workers [9–17,28]. It is now generally admitted that the rate-controlling step is the germination of massive nuclei, whereas the subsequent development of massive grains occurs very rapidly [3,5]. However, massive transformation is clearly a time-dependent phenomenon. Caretti et al. [20] performed continuous cooling experiments on several metallic systems and found that increasing the cooling rate leads to a decrease in the massive transformation temperature, an increase in the nucleation density and a refinement of the final microstructure. It has also been established that nucleation of massive domains predominantly occurs at heterogeneous sites (grain boundaries and defects). However, the existence of specific orientation relationships between massive nuclei and parent grains has been a subject of controversy. Several theories were put forward regarding the mechanisms of massive grain nucleation and these have been compared with experiment findings. It was concluded that a viable nucleus forming at a parent–grain boundary should have a low-energy facet with one of the parent grains in order to minimize the activation

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energy of nucleation [11]. Mechanisms are far from being established for growth of massive domains. The moving interface between the growing massive grain and the host grain was often reported or considered to be incoherent. The nature of the incoherent interphase interfaces, especially during massive transformation, was discussed by Massalski et al. [8] and was debated in a session during the TMS Hume–Rothery Symposium in 2004 [18]. Yanar et al. [21] and Howe et al. [22] have done some experiments using high-resolution transmission electron microscopy (TEM) on growing massive interfaces. They concluded that though these massive interfaces consist of prominent facets along various length scales, they exhibit no specific orientation relationship with the matrix inside which it is growing. However, a few authors observed that certain dense planes of the two phases involved in the massive transformation could sometimes be nearly parallel [11,19].

Massive transformation is also observed in near-γ TiAl alloys, when rapidly transformed from the high-temperature α solid solution. It was confirmed that the transformation develops with no composition change, but involves short-range atomic jumps across the moving γ/α interface [25]. In the case of continuous cooling, this transformation mode appears intermediate between modes controlled by long-range diffusion, as lamellar, Widmanstätten and "feathery" transformations (occurring at lower cooling rates), and the direct $\alpha \rightarrow \alpha_2$ ordering transformation (higher cooling rates). Consequently, γ-massive (hereafter noted γ_m) colonies are often observed coexisting with other types of microstructures. Veeraraghavan et al. [24] measured the transformation temperatures and growth kinetics of massive transformation in several TiAl alloys. Hu et al. [34] located the domains of the different transformation modes as a function of cooling rate for TiAl alloys with different compositions. They showed that for some alloys there is an optimum cooling rate for which a 100% massive transformation takes place. Wittig [23] was able to arrest the initial stages of heterogeneous nucleation and growth and found that a γ nucleus forming along the α/α grain boundaries often follows a specific orientation relationship with one of the neighboring α grains, i.e., $\{111\}_\gamma//(0001)_\alpha$ and $\langle 110\rangle_\gamma//\langle 11\bar{2}0\rangle_\alpha$. This relationship, known as the Blackburn orientation relationship (BOR) [35], is very usual in TiAl alloys. Indeed, it is also observed between γ and α_2 phases developing through lamellar, Widmanstätten or feathery transformation modes [36–38]. Dey et al. [31] used electron backscatter diffraction (EBSD) to study the heterogeneous nucleation of γ_m nuclei along α/α boundaries. They confirmed that BOR is systematic, but they also noted that not all α/α grain boundaries give rise to γ_m nuclei. Intragranular γ_m nucleation was also reported by some authors [23], but a careful examination of the nuclei shows that the nucleation is heterogeneous and occurs on twin boundaries present inside the α grain [31].

Veeraraghavan et al. [26] calculated the critical free energy (ΔG^*) associated with γ_m nuclei forming at grain boundaries, triple junctions or quadruple points (denoted as grain face, edge and corner sites in Ref. [26]). They found that sufficiently low ΔG^* values can be expected for single-faceted (having one low-energy interface) grain boundary nucleation as well as for non-faceted and incoherent nucleation at grain edges and corners. In counterpart, formation of non-faceted nuclei along grain faces and formation of faceted nucleus with growth on both the sides of the grain boundary is unexpected. However, these authors observed γ_m nuclei, which deviate from the BOR with respect to its parent α grain, for both grain face and edge nucleation. Similar results have also been reported by Wang et al. [25] while analyzing orientation maps and TEM micrographs.

The purpose of the present work was to take a closer and more statistical look at the γ-massive nucleation in a near-γ TiAl alloy, using an EBSD approach on large surfaces. The objectives were to identify the preferred sites for the formation of the nuclei and to analyze the orientation relationships between nuclei and host α grains, in order to determine if some local crystallographic configurations appear favorable for γ_m nucleation. In the following, the results obtained will be described and analyzed. Nucleation and growth mechanisms will be proposed to explain the variant selection observed.

2. Materials and methods

The material under study was a Ti–45Al–7Nb–0.2C (at.%) alloy. A sample of about $6 \times 6 \times 10\ mm^3$ was cut from a cylinder prepared by conventional casting followed by high-temperature extrusion. This sample was heat treated up to 1350 °C at a rate of 5 °C min^{-1}. It was held at 1350 °C (α phase domain [39]) for 15 min under a constant flow of argon, and then quenched in cold brine water (~ -10 °C). This rapid cooling resulted in a microstructure containing small amounts of γ-massive (γ_m) phase accompanied with coarse α_2 grains.

The specimen was mechanically and electrolytically polished, using a solution of 10% perchloric acid (70% pure) in methanol, at 10 °C and 17 V. Scanning electron microscopy (SEM) in a JEOL JSM-6500F equipped with a field emission gun (FEG) was used for the microstructure observation. Images of the microstructure were taken in backscattered electron (BSE) mode.

Crystallographic information about α_2 and γ_m phases was obtained by electron backscatter diffraction (EBSD), using Channel 5 software from HKL. In order to achieve a statistically relevant analysis of the γ_m nucleation sites, one very large orientation image mapping (OIM) was acquired over an area of $5 \times 3\ mm^2$, with a step size of 4.5 μm. Since this step size was large, compromise had to be made with the resolution. In order not to miss any grain boundaries and triple junctions with the γ_m domains, BSE images were also taken in the same area and used as reference. α-Grains with an area less than 40 μm^2 were ignored. Several other OIMs were also measured on particular grain