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The displacive-diffusive formation of single-tilt surface reliefs produced by precipitation of γ lamellae in Ti48Al2Cr2Nb alloy

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ABSTRACT

In order to give an insight into the formation mechanism of γ lamellae during α to γ phase transformation, surface reliefs produced by precipitation of γ lamellae on pre-polished specimen surface in Ti48Al2Cr2Nb alloy were systematically investigated. The results show that the surface reliefs are of single-tilt type and terraces and step risers can be observed on the surface relief. The height of the surface relief is closely related to the width of the γ lamella and each surface relief of regular γ lamellae consists of γ lamella and accommodated matrix. Terraces of the surface relief actually have needle-tooth shaped interfaces in atomic scale and step risers consist of nano-sized steps. These indicate that the initial stage of the precipitation of γ lamellae involves in a shear process and the increase in width of γ lamellae is achieved in a diffusional process by the motion of growth steps. The shear and diffusional processes finally induce an accommodation of the matrix near the specimen surface, resulting in the formation of abnormal structure layer and surface relief.

1. Introduction

The γ-TiAl based alloys with lamellar structure have attracted special attention for high-temperature applications in recent years owing to their excellent properties [1–3]. The mechanical properties of the alloys have been found to be strongly sensitive to the parameters of lamellar microstructure and the transformation mechanism during the formation of lamellar structure in γ-TiAl based alloys has been considered to be of prime importance to improve the properties at ambient temperature and high temperature [4–6]. The lamellar structure consists of γ lamellae (or plates) with face-centered tetragonal (fct) $L1_0$ structure and alternate α_2 lamellae (or plates) with hexagonal close-packed (hcp) $D0_{19}$ structure [7,8]. It is now generally acknowledged that γ lamellae and α_2 lamellae are formed from the high-temperature disordered α phase with hcp A3 structure by precipitation reaction of α to γ transformation and ordering reaction of α to α_2 transformation, respectively [9]. Transformation mechanism involved in the formation of γ lamellae has been extensively studied and three different types of transformation mechanisms, displacive, diffusional and displacive-diffusive, have been established [10–13]. Therefore, the conflict about the transformation mechanism of the formation of γ lamellae still exists and more experimental evidences are needed.

It has been accepted that surface reliefs are closely related to the transformation mechanism in alloys, and the observations of surface reliefs and the fine structure on it by using atomic force microscopy (AFM) have substantially improved the understanding of transformation mechanism of phase transformation involved in steels [14]. Recently, AFM has been used to characterize surface reliefs and fine structures on surface reliefs (such as step risers, terrace surfaces and kinks) resulting from the precipitation of γ lamellae from high-temperature disordered α phase, and thus gives an insight into the transformation mechanism of the γ lamellae [15]. In γ-TiAl based alloys, the surface relief was firstly noticed by Valencia et al. on the dendrite arms of the prior α phase, and then qualitatively examined by Sun and regarded as an evidence of invariant-plane-strain (IPS) type transformation for the precipitation of γ lamellae [16,17]. Sun explained the α to γ transformation with a shear mechanism based on the observation of this IPS-type surface relief. Then, J. F. Nie et al. pointed out that IPS-type surface relief could also be formed by a diffusional mechanism which may be first proposed by Y. C. Liu and H. I. Aaronson [18,19]. Although many investigations have been conducted on the characteristics and formation of surface relief in γ-TiAl based alloys, to our knowledge, there is no work relating the detailed atomic arrangement and interfacial structure of surface relief and the adjacent matrix to the transformation mechanism of γ lamellae in γ-TiAl based alloys. Moreover, some experimental evidences have revealed that the crystallographic

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features of the precipitate near the specimen surface are different from that in the sample interior [20]. Therefore, the microstructure from the specimen surface to the areas well below it should be examined by using special sample preparation methods, such as Focused Ion Beam (FIB) technology.

In this work, the fine structures of surface relief produced by the formation of γ lamellae, the atomic arrangement and the interfacial structure between surface relief and its adjacent α_2 matrix from specimen surface to the interior were exactly investigated by AFM and Transmission electron microscopy (TEM). The purpose of the present study is to inspect the essential characteristics of surface relief induced by the precipitation of γ lamellae and thus to reveal the transformation mechanism controlling the formation of γ lamellae.

2. Materials and methods

The Ti48Al2Cr2Nb (composition in atomic percent, at. %) alloy used in this study was prepared by vacuum arc melting process. Specimens with 6 mm in diameter and 3.5 mm in thickness were cut from the ingot and then mechanically polished by standard metallographic techniques to achieve a smooth top surface. The parallelism between the top and bottom surfaces of the specimens was carefully controlled. After that, the specimens were put into a VL200DX-SVF17SP high-temperature laser scanning confocal microscopy (HTLSCM) in a high purity argon atmosphere, then were heated to 1430 °C in the single α phase field region of this alloy at a rate of 1.67 °C/s and held for 5 min. Subsequently, the specimens were cooled at different cooling rates from 0.05 °C/s to 2 °C/s to form lamellar microstructure.

The growth process of γ lamellae was in-situ observed on the top surfaces of the specimens and recorded by HTLSCM. The examination of these as-prepared top surfaces was directly conducted at ambient temperature using scanning electron microscopy (SEM) on a Tescan MIRA 3 XMU. The surface reliefs induced by precipitation of γ lamellae were characterized by AFM on NT-MDT equipment. TEM foils were prepared from the top section of the specimens by using a Tescan GAIA3 dual beam FIB system. Conventional and high-resolution TEM (HRTEM) investigations of the FIB foils were performed on an FEI Tecnai G^2 F30 microscope with an accelerating voltage of 300 kV. All TEM images were obtained with the incidence beam along the $[11\bar{2}0]_{\alpha_2}$ zone axis, unless specified otherwise.

3. Results and discussion

3.1. Microstructure produced by precipitation of γ lamellae

When the Ti48Al2Cr2Nb alloy was cooled down from single α phase filed with low cooling rates, γ lamellae would precipitate from the high-temperature disordered α phase [21]. This process was observed by using HTLSCM and a typical result is shown in Fig. 1(a). The long thin strips with dark contrast are γ lamellar precipitates and the areas with bright contrast are the untransformed α matrix. At ambient temperature, the microstructure with dark and bright contrast strips was examined under SEM and the result is presented in Fig. 1(b). The microstructure can be found to consist of lamellae with different width in this SEM-secondary electron (SE) image. The fine lamellae (FL) have an average width of a fraction of a micron and the regular lamellae (RL) have a width of about 1–3 μm, which agrees with the results in a previous investigation [22]. Occasionally, there are some abnormal lamellae (AL) with a width of about 7–9 μm formed at the lowest cooling rate. It is interesting to find that there are fine undulations on the lamellae and gullies between the lamellae. A three-dimensional (3-D) representation of the area near AB in Fig. 1(b) is presented in Fig. 1(c). It can be found that each regular lamella corresponds to a single-tilt surface relief [23] and the single-tilt surface reliefs have same incline direction in one prior α grain. The undulations structures of surface reliefs in Fig. 1(b) are terraces and step risers, which show rounded corners or a more rectangular shape in most cases. They are defined as transformation disconnections (TD) or structural disconnections (SD) by Aaronson et al. [24]. This indicates a diffusional ledge-wise mechanism is followed during the α to γ transformation. The presence of rounded corners demonstrates that the terraces grow from the side where inclined surface forms and the width of the lamella increases. Topographic line-scan along CD, which is chosen to be perpendicular to the lamella, was conducted and the profile curve is presented in Fig. 1(d) with the measured tilt angles. The curve demonstrates that the single-tilt surface relief suddenly protrudes from the specimen surface with a height of about 300 nm, leading to the formation of an inclined surface. The height of the single-tilt surface relief is reduced through a skew-line segment which corresponds to the terrace surface and a polyline segments which corresponds to the alternation of step risers and terrace surfaces. Obviously, this outlet of the surface relief has more regularity than that produced by bainitic transformation [14]. The values of tilt angle of the surface relief can be found to vary along the profile, implying the surface relief is not of an invariant plane strain (IPS) type according to the previous work [19]. The inclined surface of the surface relief indicates that the formed γ lamellae have a shear or displacive nature, which may be a result of a continuous activity of Shockley partial dislocations with same Burger vector to maintain the lattice correspondence during the α to γ phase transformation [18]. Whereas the step risers and terraces reveal the existence of diffusive characteristic in this transformation [19,23,25]. To investigate the essential characteristics of these surface reliefs, a TEM foil was made along AB and perpendicular to the top surface of the specimen by using FIB technique, as shown in the inset in Fig. 1(b). It can be seen that the outline of the upper edge of the TEM foil exactly matches the 3-D result in Fig. 1(c).

3.2. Phase constitution of the surface relief

The TEM foil shown in the inset of Fig. 1(b) was examined and the bright-field (BF) and the corresponding dark field (DF) images are shown in Fig. 2(a) and (b), respectively. The selected area electron diffraction (SAED) pattern shown in the inset of Fig. 2(b) was taken from the area in the interior of the TEM foil, as marked by a white circle in Fig. 2(b), which is indicative of the Blackburn's orientation relationship (BOR) between the γ lamella and the α_2 matrix [21]. The DF image in Fig. 2(b) was obtained by using the superlattice reflection $(1\bar{1}00)_{\alpha_2}$, as indicated by the yellow arrow shown in the inset of Fig. 2(b). It clearly shows that the most part of a single-tilt surface relief is composed of γ lamella, whereas the bottom part of the inclined surface is composed of α_2 lamella. In addition, the surface reliefs of FL (the left part in Fig. 2(b)) is found to be very slight, while those of the RL (the right part in Fig. 2(b)) are salient. It indicates that the width of the γ lamella is of key importance in the production of surface relief. In other words, the precipitation of wider γ lamella induces the formation of more prominent surface relief. Fig. 2(c) shows a magnified image of the area near the single-tilt surface relief produced by RL in Fig. 2(b). The single-tilt surface relief is observed to reach its maximum height from the side which has an inclined surface, and then its height gradually decreases to fit the height of the matrix on the other side, which is consistent with the profile curve in Fig. 1(d). The original position of specimen surface is marked by the dotted line, and zones of γ phase and α_2 phase are labeled according to the result in DF image in Fig. 2(b). From Fig. 2(c), it is interesting to note that a change of image contrast is found near the specimen surface except for the area of the inclined surface. In order to further investigate the detailed structural characters of the surface relief and the change of image contrast near specimen surface, HRTEM experiments were carried out to comprehend the atomic arrangement of both the γ precipitate and α_2 matrix and to explore whether there is a difference in orientation relationship (OR) between these two phases in the areas near and well below the specimen surface. The areas examined by HRTEM are illustrated by squares