Acta Materialia 96 (2015) 352–365

Contents lists available at ScienceDirect

Acta Materialia

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

Control of γ lamella precipitation in Ti–39 at.% Al single crystals by nanogroove-induced dislocation bands

Dai-Xiu Wei [a,b], Yuichiro Koizumi [a,*], Akinori Yamanaka [c], Masahiko Yoshino [d], Yungping Li [a], Akihiko Chiba [a]

[a] Institute for Materials Research, Tohoku University, 2-1-1 Katahira, Sendai, Miyagi 980-8577, Japan
[b] Department of Materials Processing, Tohoku University, 6-6-02 Aramaki Aza Aoba, Sendai, Miyagi 980-8579, Japan
[c] Department of Mechanical Systems Engineering, Tokyo University of Agriculture and Technology, 2-24-16 Nakacho, Koganei, Tokyo 184-8588, Japan
[d] Graduate School of Science and Engineering, Yamagata University, 4-3-16 Jonan, Yonezawa, Yamagata 992-8510, Japan

ARTICLE INFO

Article history:
Received 30 January 2015
Revised 5 May 2015
Accepted 13 May 2015
Available online 25 June 2015

Keywords:
Titanium aluminide
Optical grating
Nanogrooving
Dislocation band
Aging

ABSTRACT

We attempted to control γ lamellae precipitation in Ti–39 at.% Al single crystals by nanogrooving process (NGP) intending to introduce periodic dislocation bands as preferential nucleation sites of γ-TiAl lamellae precipitates. In NGP, grooves were formed along the trace of (0001) basal plane on the surfaces of single crystal of α_2-phase supersaturated with Al-atoms by nano-scale plastic deformation using diamond knives with the edge angle of 60°. The widths of grooves ranged from 180 nm to 1860 nm for the load per knife-edge length in the range from 0.3 N/mm to 8.3 N/mm. Beneath the grooves, dislocation bands consisting of both basal dislocations and non-basal dislocations were formed. After aging at 1073 K for 1×10^4 s, pairs of γ lamellae were found to form only beneath the grooves to depths greater than 30 μm, meaning that the location of γ lamellae could be controlled by NGP. Increasing the aging temperature to 1173 K provided even greater control, with arrays of γ lamella bundles exhibiting a periodicity identical to that of the nanogrooves, and even near the surface no γ lamellae were formed in un-deformed regions. No concentration gradient was found within individual γ lamellae, which had a near-equilibrium Al concentration; thinner γ lamellae tended to have a slightly lower Al concentration. These results indicate that the precipitation of γ lamellae can be effectively controlled by introducing periodic dislocation bands using NGP, which opens new avenue for the manufacturing of near- or sub-wavelength grating structures with ultra-high aspect ratios through the selective dissolution of these γ lamellae.

1. Introduction

Near- and sub-wavelength gratings with a periodicity in the range of 100 nm to 1 μm [1] have attracted considerable interest in recent years as important components in optical devices owing to their excellent polarization-independent diffraction [2], isolation [3], and phase control [4]. This has seen them widely used in optical information processing, filters, polarization beam splitters, sensors and optical isolators [5–7,3]; i.e., applications in which the optical characteristics of the grating are determined by their period, line width and depth. At present, these grating structures are mainly fabricated by lithography-based technologies. Recently, mask-less processes are attracting attention. For instance, scanning near-field photolithography (SNP) with laser coupled near-field scanning optical microscopy (NSOM), which is capable of achieving a period of 100 nm and a width of 45 nm [8,9]. Focused ion beam (FIB) etching presents an alternative mask-less process, whereby nanometer-scale gratings can be directly fabricated on very hard materials such as silicon [10], and has achieved high aspect ratio gratings with a pitch of 500 nm and depth of 2 μm on poly-methyl-methacrylate. Silicon gratings with a period of 666 nm and depth of 200 nm have also been produced by X-ray lithography and electron beam lithography (EBL) combined with fast atom beam etching (FAB) [11].

Despite the success achieved with existing techniques, there is a demand for new processes that are productive and less costly than lithography based methods. This has led to nano-imprint lithography (NIL) becoming increasingly popular, as it represents a low-cost and high-throughput method of fabricating sub-wavelength grating structures on polymers, metals and glass when combined with reactive ion etching [12–14]; however, the mold needed for this is quite costly and inflexible.

* Corresponding author.

http://dx.doi.org/10.1016/j.actamat.2015.05.016
1359-6454/

The microstructures of Ti–Al alloys have been investigated extensively with special focus on their effects on the mechanical properties for its attractive characteristics as light-weight high-temperature structural material [15]. But we have considerable interest in producing gratings from the lamellar structure of α_2-Ti_3Al (hexagonal close packed (hcp)-based $D0_{19}$-type ordered structure) and γ-TiAl (face centered close-packed (fcc)-based $L1_0$-type ordered structure), which can be created through the precipitation of γ-TiAl plates from a supersaturated α_2-Ti_3Al matrix by aging at the $\alpha_2 + \gamma$ dual phase temperature [16–20].

The orientation of the precipitated γ lamellae relative to the α_2 matrix, otherwise known as Blackburn's relation [17], is expressed as:

$$(0001)_{\alpha_2}//\{111\}_{\gamma}$$

$$\langle 11\bar{2}0\rangle_{\alpha_2}//\langle 1\bar{1}0\rangle_{\gamma}$$

Note that very flat interfaces are formed parallel to the (0001) basal plane of the α_2 matrix and the {111} plane of the γ phase. Moreover, the thickness of the γ lamellae can be adjusted by changing the time and temperature of aging [18,19], but this does not ensure an even lamellar spacing or distribution. The difference in polarization behavior between the α_2 matrix and γ phase is significant, as it means that the γ lamellae can be selectively dissolved from single crystals by electrochemical dissolution to create nano-scale grating structures consisting of three-dimensional α_2-Ti_3Al lamellae and crevasses [21,22]. However, the period and width of such a grating can only be made homogeneous if the initial lamellar structure can be controlled.

In our previous study [23], it was indicated that the precipitation of γ lamellae from an Al-supersaturated α_2 matrix is accelerated by plastic deformation prior to aging. This was mainly attributed to the effect of introduced dislocations, with both basal and prismatic dislocations in Al-supersaturated α_2-Ti_3Al single crystals accelerating the precipitation of γ lamellae by acting as preferential nucleation sites for γ plates. It was also found that basal dislocations are more effective, even though they have an identical Burgers vector to that of prism dislocations [23]. This means that the localized introduction of basal dislocations could conceivably allow the precipitation and distribution of γ lamellae to be precisely controlled. Furthermore, if basal dislocations could be introduced in regions as narrow as an individual γ lamella, it should be possible to manipulate every single lamella to create a grating structure with well-defined dimensions through aging and selective dissolution.

The nano plastic forming (NPF) technology developed by Yoshino et al. [24–28] makes it possible to induce plastic stain within well-controlled regions of a metal's surface ranging in size from 10 nm to microns. This nanogrooving process (NGP) uses a diamond knife tool and piezo-driven stage to create an array of nano-grooves on the surface of a metal as little as 10 nm apart. A previous study [29] has revealed that applying this process to a Ti–39 at.% Al single crystal with a load of 5 N introduces narrow (<1 μm wide) dislocation bands consisting of both basal and non-basal dislocations that extend to a depth of several micrometers beneath each groove. These cause pairs of very long and thin γ lamellae to be formed beneath each groove upon aging, though some short γ lamellae are also formed in regions without plastic deformation. Thus, although the precipitation of γ lamellae is accelerated by the dislocation bands, it is not as well controlled as desired. That said, if the appropriate parameters needed to control γ lamella precipitation can be determined, then it should be possible to achieve sub-wavelength gratings with a super-high aspect ratio through a combination of NGP, aging, and selective dissolution of γ lamellae from Ti–Al single crystals [21,22].

Based on the findings of previous studies [21–23,29], manipulating the precipitation of γ lamellae through the localized introduction of strain seems the most viable approach to achieving a low cost, high-volume process for producing high aspect ratio gratings. This study therefore uses NGP to induce plastic strain within nanometer-scale regions of Ti–39 at.% Al single crystals to create parallel basal dislocation bands with a relatively high dislocation density. The effects of varying the conditions used for NGP and subsequent aging are herein discussed with a view to determining the optimal approach to create sub-wavelength gratings.

2. Experimental procedures

2.1. Sample preparation

A rod of Ti–39 at.% Al alloy measuring 10 mm in diameter and 150 mm in length was produced by melting commercially pure (99.9%) Ti and high purity (99.99%) Al in an arc melting furnace. This melting process was repeated four times in order to ensure a homogeneous composition, and then a floating-zone (FZ) method was applied to produce a single-crystal ingot under a high-purity Ar gas atmosphere at a growth rate of 2 mm h^{-1}. The resulting crystal was sealed in a quartz capsule with high-purity Ar gas and homogenized at 1273 K for 72 h, after which it was sliced parallel to the (0001) plane of the α_2-Ti_3Al phase into pieces 2.5 mm thick. These pieces were also sealed in capsules under high-purity Ar and subsequently aged at 1473 K for 8.6×10^4 s to obtain a single α phase. Finally the capsules were broken open in water quickly and the pieces water-quenched to produce a supersaturated α_2-Ti_3Al single crystal with a nominal composition of Ti–39 at.% Al.

These crystals produced were cut into plates measuring 2.5 mm in length, 2.5 mm in width and 0.5 mm in thickness, with the normal direction being set parallel to the (0001) basal plane of the α_2 phase, as illustrated in Fig. 1a. In other words, the (0001) basal plane of the supersaturated α_2 phase was aligned perpendicular to the sample surface on which NGP was to be conducted. This was intended to allow γ lamellae to be produced perpendicular to the surface based on Blackburn's orientation relationship. The plates were ground and wet polished with 1200-grit abrasive paper to remove any oxide film produced by water quenching, and then polished to a mirror finish with colloidal silica (OP-S). After degreasing by sonication in acetone, the plates were subjected to NGP by loading along the $[7\bar{2}\bar{5}0]$ direction (Fig. 1b). A surface-relief grating structure was then obtained by aging and subsequent selective dissolution of the γ lamellae.

2.2. Nanogrooving process

The nanogrooving process was conducted in a clean booth at room temperature, with a knife edge angle of 60° and edge radius of less than 50 nm being used (Fig. 1a). Two different knife edge lengths were used (1.5 and 0.6 mm), though in both cases the knife was driven by a piezoelectric-actuator to create individual grooves on the crystals' surface at loads of 0.5, 1, 2, 3, 4 and 5 N. An interval of 40 μm was set between each groove to better observe the formation of individual γ lamellae, but this was later reduced to 2 μm to obtain a near-wavelength grating structure. With both scenarios, an array of ten grooves was produced as a single group for each load, giving six groups on each crystal. To precipitate γ lamellae after NGP, the crystals were sealed in quartz capsules with high-purity argon gas and aged at 1073 K (for 1×10^4 s) or 1173 K (for 1×10^4 and 1×10^5 s). The capsules were then broken open in water quickly and the crystals water quenched.