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Dislocations in Gadolinium Gallium Garnet ($Gd_3Ga_5O_{12}$): III. Nature of Prismatic Loops and Helical Dislocations

Abstract: Thin garnet films suitable for magnetic bubble devices can be made by depositing the film material onto nonmagnetic garnets such as $Gd_3Ga_5O_{12}$ (GGG). The performance of these devices is influenced by the dislocation content of the films. This, in turn, depends on the dislocation content of the substrate. Dislocations in the substrate can be detected by means of the birefringence they induce, or from the etch pits formed where they meet the sample surface. Most of the dislocations revealed by these techniques have been climb loops around inclusions and helical dislocations. This paper describes an optical method for determining the sign of the stresses at inclusions and nature of loops and helical dislocations. The method has shown that iridium inclusions are compressed by the matrix and that the loops and helices in GGG are extrinsic; they grow either by the emission of vacancies or the absorption of interstial atoms.

Introduction

Single crystals of gadolinium gallium garnet ($Gd_3Ga_5O_{12}$ or GGG) are used as substrates for magnetic garnet films employed in bubble domain devices [1,2,3]. The motion of the magnetic domains in these devices is impeded by dislocations in the films. The most important sources of dislocations in the films are dislocations in the substrate. The dislocations in the films are, in the majority of cases, simply extensions of dislocations that terminated on the substrate surface before film growth began [4,5]. Thus, an essential step in the elimination of dislocations from magnetic garnet films is the preparation of dislocation-free substrates.

The aim of the series of papers [6,7] of which this is Part III is to describe the geometry, the formation, and finally the avoidance of dislocations in GGG crystals grown by the Czochralski technique. Part I [6] described loops around iridium inclusions. The loops were formed by a climb mechanism and were much larger than the inclusions responsible for them. The Burgers vectors of the loops were parallel to ⟨111⟩, ⟨110⟩ or ⟨100⟩. Part II described large helical dislocations. The Burgers vectors of the helices were parallel to ⟨111⟩ or ⟨100⟩. In Part I, it was stated that the iridium inclusions were compressed by the garnet matrix and that the climb loops grew either by the absorption of interstitial atoms or the emission of vacancies. In Part II, it was stated that the helical dislocations in GGG grew by the absorption of interstitial atoms or the emission of vacancies. However, the evidence on which these statements were based was not given. This paper presents that evidence. Experiments show that the iridium inclusions were compressed by the garnet matrix, and that the loops and helical dislocations were interstitial or extrinsic. The experimental method used combines the birefringence technique of Bond and Andrus [8] with the optical system of Kear and Pratt [9]. It allows one to observe individual dislocations and to determine the signs of the stresses associated with them.

Experimental details

Wafers of GGG were cut from cylindrical boules grown by the Czochralski technique. The growth direction was [111] and an iridium crucible was used to contain the melt. Most wafers were oriented with (111) parallel to their planes. The remainder were {110}, {100}, or {112} wafers and were prepared to permit viewing of the defects along ⟨110⟩, ⟨100⟩ and ⟨112⟩. All wafers were carefully polished to produce clean and scratch-free surfaces. Some of the polished (111) wafers were etched in a hot (150 to 200°C) solvent prepared by mixing equal volumes of concentrated sulphuric and phosphoric acids. This treatment resulted in the formation of triangular etch pits at the emergence points of some but not all the dislocations present.

All wafers were examined in a polarizing microscope with polarizer and analyzer set at right angles to each other. A sensitive tint plate was incorporated in the optical system [9]. This device is a full-wave plate for light with wavelength near the center of the visible spectrum. It is inserted between the specimen and the analyzer with its optic axis inclined at 45° to the polarizer and the analyzer. In this setting, and in the absence of a birefringent specimen, the light transmitted to the eye from a white source is a mixture of red and blue. If a garnet wafer containing elastic strain gradients due, for example, to dislocations, inclusions, or changes in chemical composition, is inserted, the image is divided into regions that are either mostly blue or mostly red. The areas that appear red or blue can be altered or interchanged by rotation of the specimen about the optic axis. The signs of the stresses present in the red and blue areas can be determined from the color changes that take place when stresses of known direction and sign are applied to the sample [9]. In the experiments described here the applied stresses were compressive. They were applied along one direction at a time and were always parallel to the wafer plane.

Observations

• *Sign of stress at iridium inclusions*

Several different inclusions have been identified in GGG [10]. The ones described here are iridium and come from the crucible used to contain the melt [6]. One property of the iridium inclusions is that they are opaque. The stresses inside them must be deduced from the optical behavior of the matrix. Another property of the inclusions is that they do not have a particular orientation relationship with the matrix—indeed, the orientations are almost random. This indicates that they are not precipitated from the solid but are trapped in it when solidification takes place. The absence of an epitaxial relationship between the inclusions and the matrix means that the interfaces between the inclusions and matrix are incoherent [6]. This in turn means that the stresses inside the inclusions are hydrostatic [11]. The majority of iridium inclusions in GGG are plate-like. A hydrostatic stress inside a plate-like inclusion gives rise to an almost uniaxial stress immediately outside its broad faces. This stress is perpendicular to the broad faces; it is compressive if the inclusion is too large for the hole in the matrix and tensile if it is too small [11].

An optical micrograph of a plate-like iridium inclusion in a GGG wafer oriented with $(11\bar{2})$ parallel to its plane is seen in Fig. 1(a). The broad faces of the platelet are perpendicular to the [111] direction in the wafer plane. If the almost uniaxial stress discussed in the previous paragraph were compressive, then the application of a compressive stress along [111] would cause the blue lobes visible outside the broad faces to grow. If the almost uniaxial stress were tensile, then a compressive stress along [111] would cause the blue lobes to shrink. The effect of a compressive stress along [111] is revealed by Fig. 1(b). It is clear that the stress caused the blue lobes to grow. The iridium inclusion was therefore compressed by the matrix. This result is consistent with the thermal expansion coefficients of iridium and GGG.

• *Nature of climb loops around inclusions*

Figure 2(a) is a micrograph of an iridium inclusion in a wafer oriented with (011) parallel to its plane. The inclusion resembles the one in Fig. 1 in that it is plate-like, is flanked by blue lobes, and its broad faces are perpendicular to the wafer plane. It differs in that it is encircled by a succession of large, concentric, climb loops [6]. These loops lie on a $(1\bar{1}1)$ plane perpendicular to the film plane and have Burgers vectors along $[1\bar{1}1]$. The loops give rise to a pair of blue streaks that lie along $[21\bar{1}]$. A careful examination of these streaks reveals small, intermittent contrast variations. These contrast variations mark the points at which individual climb loops cross the plane whose image is in sharp focus. The fact that the climb loops and the lobes outside the broad faces of the inclusion in Fig. 2(a) have the same color shows that the stresses normal to the inclusion and normal to the climb loops have the same sign. This, taken together with the result given in the preceding subsection, suggests that the climb loops are interstitial or extrinsic. Confirmation of this is provided by Fig. 2(b), which shows the effect of a compressive stress applied along $[1\bar{1}1]$. It can be seen that this stress caused the blue lobes outside the inclusion, and the blue streaks due to the climb loops, to grow.

The formation of interstitial loops at compressed inclusions is, at first sight, a rather surprising result. It indicates that an inclusion which is already too large for the hole available to it promotes the precipitation of additional material. An explanation is provided by Mott and Nabarro's [11,12] determination of the stress field of an inclusion. The explanation holds for inclusions of all shapes but is easiest to visualize for spherical ones. The stress outside a spherical inclusion is a shear without dilation [12]. If the inclusion is too large for the hole in the matrix, then the radial stress in the matrix is compressive, and the stress parallel to the inclusion surface is tensile. It is this tensile stress that promotes the nucleation and growth of interstitial climb loops. The conditions that lead to nucleation are discussed by Matthews, et al. [5,6,13,14]. They find that nucleation is improbable unless the concentration of the appropriate point defect is very different from the equilibrium value.

Figure 1 (Facing page, top) (a) Optical micrograph (×360) of a plate-like iridium inclusion in a garnet wafer oriented with $(11\bar{2})$ parallel to its plane. The plane of the platelet is approximately perpendicular to [111]. The blue lobes outside the broad faces of the inclusion are due to elastic stresses generated by the inclusion. (b) Micrograph of the same area taken after the application of a compressive stress along [111].

Figure 2 (Facing page, center) (a) Micrograph (×100) of an iridium platelet in a wafer oriented with (011) parallel to its plane. The platelet is encircled by a number of large, concentric climb loops on $(1\bar{1}1)$. (b) Micrograph of the same area taken after the application of a compressive stress along $[1\bar{1}1]$.

Figure 3 (Facing page, bottom) Micrographs (×100) of a helical dislocation (a) before and (b) after the application of a compressive stress along $[1\bar{1}1]$. The asymmetric color distribution is the result of a stress gradient that is not due to the helical dislocation.

• *Nature of helical dislocations*

Figure 3(a) is a micrograph of a large helical dislocation with axis and Burgers vector parallel to $[1\bar{1}1]$ in a wafer oriented with (011) parallel to its plane. A blue region is seen to extend part of the way down the axis of the helix. Careful examination of the boundary between this blue region and the red reveals that the individual turns of the helix are blue. If the helices in GGG are of interstitial type, then a compressive stress applied along $[1\bar{1}1]$ shown in Fig. 3(a) would be expected to cause the blue region within the helix to intensify and expand. On the other hand, if the helices in GGG are of vacancy type then a compressive stress applied along $[1\bar{1}1]$ would cause the blue region to contract. Figure 3(b) shows that the first of these alternatives is correct. The helix in Fig. 3 was therefore of interstitial type and grew either by the absorption of interstitial atoms or the emission of vacancies.

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