Dislocations in YAG Phase of a Directionally Solidified $\text{Al}_2\text{O}_3/\text{YAG}$ Deformed at Room and High Temperatures

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Dislocation structures of the YAG (yttrium aluminum garnet, $\text{Y}_3\text{Al}_5\text{O}_{12}$) phase in a directionally solidified $\text{Al}_2\text{O}_3/\text{YAG}$ composite were investigated by transmission electron microscopy. In the as-grown specimen, dislocation networks composed of dislocations with Burgers vectors parallel to $\langle 100 \rangle$, $\langle 110 \rangle$ and $\langle 111 \rangle$ were observed. In a specimen deformed at 1773 K, both dislocation networks and isolated dislocations with Burgers vector of $a\langle 100 \rangle$ and $a/2\langle 111 \rangle$ were observed but neither of them were glide dislocations. In a specimen deformed at room temperature, isolated dislocations with a Burgers vector of $a/2\langle 111 \rangle$ were observed. They were identified as glide dislocations lying on $\{110\}$ planes.

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I. Introduction

A directionally solidified $\text{Al}_2\text{O}_3/\text{YAG}$ eutectic ceramic (hereinafter denoted by DS- $\text{Al}_2\text{O}_3/\text{YAG}$ composite) was developed by Waku⁽¹⁾ by controlling the solidification process of an eutectic $\text{Al}_2\text{O}_3/\text{YAG}$ originally reported by Viechnicki⁽²⁾. A DS- $\text{Al}_2\text{O}_3/\text{YAG}$ composite has an excellent strength at high temperature⁽¹⁾⁽³⁾⁽⁴⁾. However, mechanism for the high strength of the DS- $\text{Al}_2\text{O}_3/\text{YAG}$ composite at high temperature is not understood well. It is composed of huge three-dimensional single crystals of Al_2O_3 and YAG⁽¹⁾⁽⁵⁾, the volume fraction of YAG phase being 45%⁽⁶⁾. Since Al_2O_3 phase has plasticity at high temperature⁽⁷⁾⁽⁸⁾, the strength of a DS- $\text{Al}_2\text{O}_3/\text{YAG}$ eutectic is likely to be controlled mainly by the strength of YAG. YAG has a chemical formula of $\text{Y}_3\text{Al}_5\text{O}_{12}$. The unit cell contains eight molecular formula unit, i.e., 160 ions. However, owing to its symmetry the structure is simple and bcc⁽⁹⁾ with space group Ia3d⁽¹⁰⁾. The aim of the present study is to analyze dislocation structures in the YAG phase of a DS- $\text{Al}_2\text{O}_3/\text{YAG}$ composite deformed at room temperature and 1773 K. Structure of grown-in dislocations was also analyzed.

II. Experimental Procedure

The specimens used in the present study were grown by one of the authors (Y. W.)⁽¹⁾⁽³⁾. They were mechanically polished first with emery papers and then with Al_2O_3 paste to the thickness of 150 μm . They were cut into rectangular pieces of 5 mm $\times$ 4 mm, the longitudinal axis being coincident with the direction of solidification.

The samples were deformed at room temperature and 1773 K by indentation. For deformation at room temperature, a standard Vickers indentation was carried out

with a load of 0.49 N and a dwell time of 5 s. At 1773 K a Brinell indentation was carried out, with a special apparatus⁽¹¹⁾⁽¹²⁾, with a load ranging from 2.94 N to 5.88 N and a dwell time of 4.32×10^4 s in air.

Foil specimens for transmission electron microscopy (TEM) observation were prepared by ion milling and a focused ion beam (FIB) technique⁽¹³⁾⁽¹⁴⁾ and examined in a JEOL-200CX microscope at an accelerating voltage of 200 kV. The nature of the dislocations was determined by a standard contrast experiment.

III. Results and Discussion

1. Structure of grown-in dislocations

Most of the grown-in dislocations in the YAG phase form sub-boundaries and few isolated dislocations exist inside a sub-grain. **Figures 1(a) to (f)** show a typical example of contrast experiments carried out on grown-in dislocations in YAG, where the dislocations were viewed along different incident beam directions (**Table 1**), i.e., $B=[001]$ ((a) to (d)), $B=[102]$ (e), $B=[112]$ (f), respectively. Figure 1(g) shows a schematic diagram of the dislocations studied. Dislocations indicated by solid lines appear in $g=080$, $g=880$, $g=4\bar{2}\bar{2}$ and $g=22\bar{2}$, while they disappear in $g=\bar{8}00$ and $g=\bar{8}08$. Thus, the Burgers vector of these dislocations is determined to be parallel to $[010]$. Dislocations indicated by railroad lines appear in $g=\bar{8}08$, $g=4\bar{2}\bar{2}$ and $g=22\bar{2}$, while they disappear in $g=080$, $g=\bar{8}00$ and $g=880$. Thus, these dislocations have Burgers vector paralleled to $[001]$. Similarly, Burgers vector of dislocations indicated by dotted lines is determined to be parallel to $[1\bar{1}1]$. Although dislocations indicated by chain lines show residual contrasts in $g=22\bar{2}$ the Burgers vector of these dislocations is parallel to $[011]$. The results of $g \cdot b$ analysis are summarized in

g & B	$g=080$ $B=[001]$	$g=\bar{8}00$ $B=[001]$	$g=880$ $B=[001]$	$g=\bar{8}08$ $B=[001]$	$g=4\bar{2}\bar{2}$ $B=[102]$	$g=22\bar{2}$ $B=[112]$	b
—	v	iv	v	iv	v	v	[010]
	iv	iv	iv	v	v	v	[001]
— · —	v	iv	v	v	v	iv	[011]
· · · · ·	v	v	iv	iv	v	v	[111]

v; visible, iv; invisible

Table 1.

Dislocation networks in the as-grown specimen are composed of dislocations with Burgers vector parallel to $\langle 100 \rangle$, $\langle 110 \rangle$ and $\langle 111 \rangle$, as schematically shown in Fig. 1(g). This is in good agreement with the result obtained on a $\text{Y}_3\text{Fe}_5\text{O}_{12}$ (YIG), a different type of garnet compound⁽¹⁵⁾.

2. Dislocation structure in a specimen deformed at 1773 K

A SEM micrograph of a DS- $\text{Al}_2\text{O}_3/\text{YAG}$ composite deformed at 1773 K is shown in Fig. 2. Cracks are observed clearly along interfaces between Al_2O_3 and YAG, as shown by arrows. In addition, within the YAG phase linear markings, which are much fainter than the cracks along $\text{Al}_2\text{O}_3/\text{YAG}$ interface, are observed, as shown by

Fig. 2 SEM micrograph showing cracks and fainter linear markings observed in a YAG phase of a directionally solidified $\text{Al}_2\text{O}_3/\text{YAG}$ deformed at 1773 K for 4.32×10^4 s.

double headed arrows. Figure 3 shows a low-magnification TEM micrograph of such fainter linear markings. Figures 4(a) and 5 show higher-magnification TEM

Fig. 3 Low-magnification TEM micrograph of a YAG grain in a directionally solidified $\text{Al}_2\text{O}_3/\text{YAG}$ deformed at 1773 K for 4.32×10^4 s.

Fig. 4 (a) Dislocation network in a specimen deformed at 1773 K. (b) Diffraction pattern from the two neighboring sub-grains 1 and 2 shown in (a).

Fig. 5 TEM micrograph showing a train of isolated dislocations in a specimen deformed at 1773 K.

m

Fig. 6 (a) to (k) TEM micrograph showing isolated dislocations in a YAG grain, deformed at 1773 K, imaged in different g vectors. The dislocations were viewed along different incident beam directions, *i.e.*, $B=[001]$ ((a) to (f)), $B=[102]$ ((g) to (i)), $B=[012]$ (j) and $B=[001]$ (k), respectively. (l) and (m) show a schematic diagram and geometry of the dislocations studied, respectively.

Fig. 7 TEM micrograph showing YAG phase deformed with Vickers indentation at room temperature. Lateral cracks (L), dislocations (D) and slip plane (S) are observed.

Fig. 8 TEM micrographs showing isolated dislocations in a YAG, deformed at room temperature, imaged in different g vectors.

Fig. 9 (a) TEM micrograph ($B=[011]$) showing a slip plane in a YAG grain deformed by Vickers indenter at room temperature. (b) shows the geometry.

micrographs. From these micrographs it is clear that the fainter linear markings are sub-boundaries composed of dislocations. The contrast of the neighboring sub-grains of YAG shown in Fig. 3 is slightly different with one another. This indicates that the orientation of these grains is slightly different from one another. This is shown more clearly in Fig. 4(a) and (b), where the electron diffraction pattern shown in (b) was taken at a sub-boundary shown in (a). Two circular Laue zones in (b) correspond to grains 1 and 2 in (a), respectively. The contrast experiment carried out on dislocation network comprising such a sub-boundary as shown in Fig. 4(b) indicates that the dislocation networks consist mostly of $b=a\langle 100 \rangle$ and $b=a/2\langle 111 \rangle$ dislocations. The feature is essentially similar to that of grown-in dislocations.

Occasionally dislocation arrays with much simpler structure, consisting of isolated dislocations, were observed, and an example is shown in Fig. 5. Figures 6(a) to (k) show results of contrast experiment carried out on the isolated dislocations shown in Fig. 5. An array of dislocations indicated by solid line in Fig. 6(l) appear in the $g=\bar{8}00$, $g=\bar{4}20$, $g=\bar{8}80$, $g=\bar{4}20$, $g=\bar{2}40$, $g=\bar{4}0\bar{2}$, $g=\bar{4}2\bar{2}$, $g=\bar{4}22$ and $g=\bar{8}80$, while they disappear in $g=\bar{0}80$ and $g=\bar{0}4\bar{2}$. Thus, the Burgers vector of these dislocations is determined to be $b=a[100]$. A dislocation indicated by dotted line appears in the $g=\bar{0}80$, $g=\bar{8}00$, $g=\bar{4}20$, $g=\bar{4}20$, $g=\bar{2}40$, $g=\bar{4}0\bar{2}$, $g=\bar{4}2\bar{2}$, $g=\bar{0}4\bar{2}$ and $g=\bar{8}80$, while it disappears in the $g=\bar{8}80$ and $g=\bar{4}22$. Thus, the Burgers vector of this dislocation is determined to be $b=a/2[\bar{1}11]$. Geometry of these $a[100]$ and $a/2[\bar{1}11]$ dislocations is shown schematically in Fig. 6(m). Since Burgers vector of these $a[100]$ dislocations is

perpendicular to the plane on which they lie, they are edge dislocations comprising a small tilt angle sub-boundary. This indicates that, at high temperature such as 1773 K, climb motion of a dislocation is predominant in YAG.

3. Dislocation structure in a specimen deformed at room temperature

Figure 7 shows a low-magnification TEM micrograph of a YAG phase in a DS- Al_2O_3 /YAG composite deformed with a Vickers indenter at room temperature. Underneath the image of the indentation many defects are observed in addition to cracks. Two types of crack are observed; one propagates along specific crystallographic planes with dislocation emission at the crack tips and the other propagates along unspecified planes without dislocation emission.

Figure 8 shows a typical example of contrast experiments carried out on those isolated dislocations which emanated from the tip of a crack. All the dislocations studied appear in $g=\bar{0}80$, $g=\bar{8}00$, $g=\bar{0}80$, $g=\bar{8}80$ and $g=\bar{4}22$, while they disappear in $g=\bar{8}80$ and $g=\bar{2}42$. This indicates that the Burgers vector of these dislocations is parallel to $[\bar{1}11]$. The dislocations shown in Fig. 7 and Fig. 8 emanate from the tip of a crack and they do not form dislocation network, so that they are likely to be glide dislocations. In order to confirm this, the specimen was tilted in such a way that the plane on which these dislocations lie is viewed end on (Fig. 9(a)). It is clear that the plane is $\{110\}$. The Burgers vector of these dislocations is included in the plane on which they lie. In other words, these dislocations are glide dislocations (Fig. 9(b)). Since $a/2\langle 111 \rangle$ is the shortest unit translation vector in bcc, dislocation with such a Burgers vector is expected to exist as a glide dislocation. Therefore, it can be concluded that the operative slip system at room temperature in the YAG phase of DS- Al_2O_3 /YAG composite is $a/2\langle 111 \rangle\{110\}$. This result is in keeping with a theoretical prediction by Rabier *et al.*⁽¹⁶⁾

IV. Conclusions

The results of observation of dislocation structures in the YAG phase of a directionally solidified Al_2O_3 /YAG composite by transmission electron microscopy are summarized as follows.

The grown-in dislocations form namely networks composed of dislocations with Burgers vector parallel to $\langle 100 \rangle$, $\langle 110 \rangle$ and $\langle 111 \rangle$. In a specimen deformed at 1773 K dislocation networks and an array of edge dislocations were observed. These dislocations have climbed and comprise a sub-boundary. In a specimen deformed at room temperature, glide dislocations belonging to an operative slip system of $a/2\langle 111 \rangle\{110\}$ are observed.

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