

# Anisotropy of electrical conductivity in dc due to intrinsic defect formation in $\alpha$ -Al<sub>2</sub>O<sub>3</sub> single crystal implanted with Mg ions



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## ABSTRACT

The electrical conductivity in  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> single crystals implanted with Mg ions in two different crystalline orientations, parallel and perpendicular to *c* axis, was investigated. The samples were implanted at room temperature with energies of 50 and 100 keV and fluences of  $1 \times 10^{15}$ ,  $5 \times 10^{15}$  and  $5 \times 10^{16}$  ions/cm<sup>2</sup>. Optical characterization reveals slight differences in the absorption bands at 6.0 and 4.2 eV, attributed to *F* type centers and Mie scattering from Mg precipitates, respectively. DC electrical measurements using the four and two-point probe methods, between 295 and 490 K, were used to characterize the electrical conductivity of the implanted area (Meshakim and Tanabe, 2001). Measurements in this temperature range indicate that: (1) the electrical conductivity is thermally activated independently of crystallographic orientation, (2) resistance values in the implanted region decrease with fluence levels, and (3) the *I*-*V* characteristic of electrical contacts in samples with perpendicular *c* axis orientation is clearly ohmic, whereas contacts are blocking in samples with parallel *c* axis. When thin layers are sequentially removed from the implanted region by immersing the sample in a hot solution of nitric and fluorhydric acids the electrical resistance increases until reaching the values of non-implanted crystal (Jheeta et al., 2006). We conclude that the enhancement in conductivity observed in the implanted regions is related to the intrinsic defects created by the implantation rather than to the implanted Mg ions (da Silva et al., 2002; Tardío et al., 2001; Tardío et al., 2008).

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## 1. Introduction

Ion implantation of crystalline solids alters only the properties of a thin layer of the near surface region. In  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> crystals, the microstructural changes due to the damage created by ion implantation have been studied in order to modify its properties, such as conductivity, optical absorption and hardness [1,2]. Particular technological importance has been placed on the huge increase in conductivity reported in  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> single crystals doped with magnesium. Analogous changes were observed when a conductive layer attributed to both doping and structural changes by ion implantation was formed near surface regions [3–5].

The increase by several orders of magnitude of the electrical conductivity in the  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> single crystals implanted with Mg with an energy value of 90 keV and a fluence of  $10^{17}$  ions/cm<sup>2</sup>, relative to the unimplanted region, was primarily associated with oxygen vacancies induced by implantation rather than with the implanted ions in a previous work [5,16]. The main purpose of this work is to

present new results relative to the anisotropy observed in the optical and electrical properties in  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> single crystals implanted with Mg ions in two different crystalline orientations, parallel and perpendicular to *c* axis. We used optical absorption measurements to oversee the bands associated with the singly-charged *F*<sup>+</sup> and the neutral *F* centers (oxygen vacancies), as these are the most common defects produced by ion implantation. The singly-charged *F*<sup>+</sup> center absorbs at 4.8, 5.4 and 6.3 eV (depending on the crystal orientation), whereas the neutral *F* center absorbs at 6.0 eV [6,7]. The electrical conductivity in samples implanted with Mg ions with the *c* axis parallel or perpendicular to the implanted face was characterized with DC electrical measurements (two-point probe method), between 295 and 490 K.

## 2. Experimental procedure

Nominally pure  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> crystals (impurity  $\approx$  100 ppm) obtained from Adolf Meller Co, with two different crystallographic orientations were used in this study:  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> single crystals of [0001] orientation (*c*-cut), with the *c* axis perpendicular to the

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implanted face, labeled as  $c_{\perp}$ , and  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> single crystals of [10 $\bar{1}$ 0] orientation ( $m$ -cut), with the  $c$  axis parallel to the implanted face, labeled as  $c_{\parallel}$ . Samples with both orientations of about 1 mm thick and rectangular shape (6 × 6 mm) were obtained from  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> single crystal disks of 20 mm diameter by cutting with a diamond saw, and subsequently, the cut edges were polished to optical quality. The samples were implanted with Mg ions at room temperature (RT) with energies of 50 and 100 keV and fluences of  $1 \times 10^{15}$ ,  $5 \times 10^{15}$  and  $5 \times 10^{16}$  ions/cm<sup>2</sup> (see Table 1 for label details). The instrument features and setup used for both optical absorption and electric characterizations are described in previous work [5].

### 3. Results and discussion

We determined the profiles for both the implanted Mg ions and the damage produced by implantation in  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> crystals with energies of 50 and 100 keV through simulations with Stopping and Range of Ions in Matter (SRIM-2006.02) [8]. The predicted ranges of the oxygen vacancies and the implanted ions are  $\approx 40$  and 60 nm and  $\approx 90$  and 110 nm, respectively.

A preliminary study of AFM images from the surface of implanted samples in both crystallographic orientations does not reveal any anisotropic morphology relative to the accumulative damage, additional to those observed in unimplanted samples, which are independent of both the implantation direction and the ion fluence.

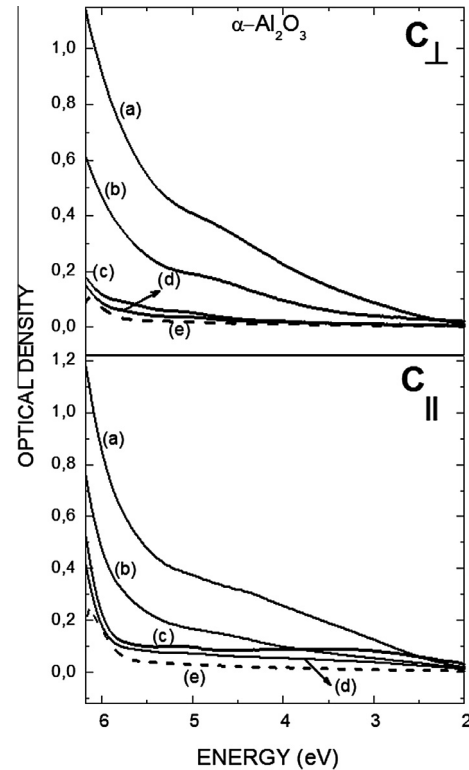
#### 3.1. Optical studies

Fig. 1 shows the polarized optical absorption spectra of the  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> samples for both crystallographic orientations,  $c_{\parallel}$  (bottom), and  $c_{\perp}$  (top) after Mg ion implantation, for the above mentioned energies and fluences. In addition the spectra before implantation for both orientations also are displayed (traces (e)). Two fundamental sets of absorption bands can be distinguished in the  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> samples after implantation, which become more apparent as the fluence and energy increase: (1) the first one is formed by two absorption bands centered at 6.0 and 5.4 eV, associated with  $F$  and  $F^+$  centers which arise from oxygen vacancy formation as a result of oxygen atoms knocked off from their lattice positions; (2) the second one is a very broad extinction band, seemingly not dependent on crystallographic orientation and centered at about 4.2 eV, which we previously attributed to Mie scattering from Mg precipitates [9,10]. Formation of metallic precipitates during implantation in oxides has formerly been observed [11,12]. In addition, although the contribution from the bands centered at 6.0 and 5.4 eV is not well resolved, their intensity increases with energy and fluence of implanted Mg ions.

**Table 1**

Sample labels, accounting for energy and fluence of implanted Mg ions, and crystal crystallographic orientation.

Crystallographic orientation	Label	Energy [keV]	Fluence [ions/cm <sup>2</sup> ]
[10 $\bar{1}$ 0] ( $m$ -cut) $c_{\parallel}$	A0	Unimplanted	
	A2	50	$1 \times 10^{15}$
	A3	50	$5 \times 10^{16}$
	A4	100	$1 \times 10^{15}$
	A6	100	$5 \times 10^{16}$
	[0001] ( $c$ -cut) $c_{\perp}$	B0	Unimplanted
B2		50	$1 \times 10^{15}$
B3		50	$5 \times 10^{16}$
B4		100	$1 \times 10^{15}$
B6		100	$5 \times 10^{16}$



**Fig. 1.** Polarized optical absorption spectra of the  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> samples implanted with Mg ions. Bottom: samples with the  $c$  axis parallel to the implanted; (a) A6, (b) A3, (c) A2 and (d) A4. Top: samples with the  $c$  axis perpendicular to the implanted; (a) B6, (b) B3, (c) B4 and (d) B2. In addition, the spectra before implantation for  $c_{\perp}$  (B0) and  $c_{\parallel}$  (A0) are shown in traces (e).

In order to reveal possible anisotropies in the formation of defects, a more clarifying picture is obtained from the difference optical spectra, as shown in Fig. 2. Each trace corresponds to the difference spectrum obtained after subtraction of the spectrum before implantation, (traces (e) in Fig. 1) from that measured after implantation (spectra a, b, c, y d in Fig. 1) for both orientations. Difference spectra show how a division can be made between samples attending to the implantation fluence:

(a) Bottom figure, corresponding to the lower fluence, presents better resolved spectra as well as a clear difference between sample orientation: while spectra corresponding to  $c_{\parallel}$  show a peak at 6.0 eV corresponding to  $F$  centers together with a broad band probably due to metal precipitates, spectra for  $c_{\perp}$  oriented samples show almost exclusively peaks at 6.3, 5.4 and 4.8 eV corresponding to  $F^+$  centers. The peak at 6.3 eV peak is at the end of the high energy region and thus poorly resolved, undoubtedly it occurs at higher energy than the one seen for  $c_{\parallel}$  implanted samples. (b) Top figure, for high fluences the above mentioned spectral features are mostly lost, and spectra are dominated by a strong broad background that points to some kind of damage in the oxygen vicinity that prevents  $F$  and  $F^+$  formation. In this case, higher implantation energies mainly result in an increase of the absorption bands intensities. The comparison of the spectra for the same type of samples implanted with different energies suggests that the formation of the previously mentioned defects, that tentatively give rise to the broad band at 4.2 eV, depends on the energy of the implanted ion as has been previously reported [5]. A similar anisotropic behavior in both defect production (mainly oxygen vacancy formation) and optical activity in the  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> single crystal implanted with Er ions has been observed [12].

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