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Electron Microscopical Investigation of Oriented Magnetite and Amphibole in Black Star Diopside

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Abstract

South Indian black star diopside contains oriented inclusions of magnetite, which are parallel to (010) of the diopside host. The magnetite blades cause the asterism effect, when the diopside is cut *en cabochon*, perpendicular to its b-axis. There are two different orientations of the magnetite inclusions, both having ‹110› of magnetite parallel to [010] of diopside.

The "Z" type inclusions have: $[1\bar{1}1]_{Mt} // [100]^*_{Di}$, while the "X" type inclusions have $[111]_{Mt} \wedge [100]^*_{Di} = 6°$. Hence these inclusions produce the fourfold asterism of star diopside. In addition, oriented amphibole inclusions are present.

Keywords: asterism, star diopside, intergrowth of diopside and magnetite, intergrowth of diopside and amphibole.

Introduction

Certain minerals when cut *en cabochon* and illuminated by a source of parallel light show asterism which is produced by scattering of the incident light by sets of oriented inclusions (WÜTHRICH and WEIBEL, 1981). In star sapphire oriented inclusions of rutile (PHILLIPS et al., 1981; SAHAMA, 1982) or of hematite (WEIBEL and WESSICKEN, 1981) cause the asterism effect. Oriented inclusions of sillimanite produce a six-fold star in star quartz (WOENSDREGT et al., 1980).

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The black star diopside from India has been the object of several gemmological studies. The aim of the present investigation is to describe the crystallographic relations between the diopside host and the inclusions causing the asterism. The identity of the inclusions has not been determined unambiguously by previous studies. A transmission electron microscope offers the best means of identifying the inclusions and studying the crystallographic relationship between star diopside and the inclusions.

According to PONAHLO (1967) the X-ray powder pattern of black star diopside recorded by an X-ray goniometer with CuKα radiation contains, in addition to the monoclinic pyroxene reflections, only magnetite reflections, which are most probably epitactically related to the pyroxene host. The presence of TiO_2 needles of less than a micron in diameter is not excluded. MARION et al. (1968) and SCHUBNEL et al. (1968) described the occurrence of magnetite lamellae showing second order dissolution of ilmenite and spinel in a South Indian star diopside. The magnetite lamellae are parallel to the faces (001) and (100) of the diopside host.

Description of the Namakkal star diopside

The star diopside sample investigated in the present study is from Namakkal (Tamilnadu), South India. The exploitation of the deposits started in about 1964. Star diopside is used by Indian women as a cheaper substitute for the more expensive star sapphire. The black star diopside from South India shows a four-fold asteriated cross, the rays of which subtend an angle of 104° (see Figure 1). The thin section cut perpendicular to the b-axis of diopside reveals, when

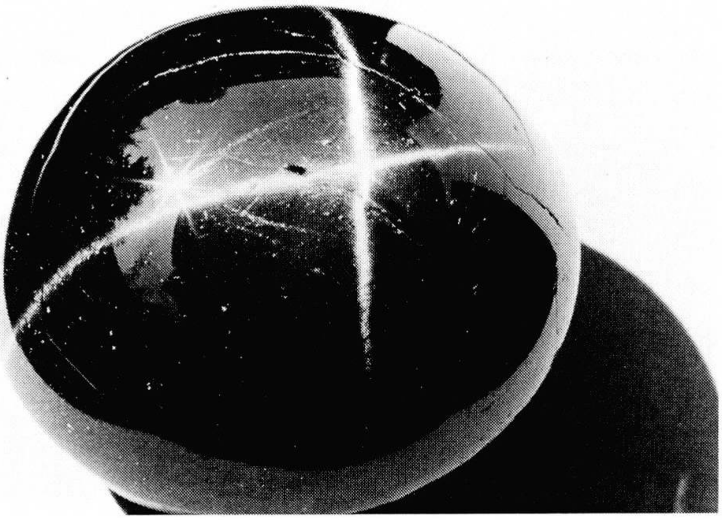

Figure 1 A cut *en cabochon* star diopside from Namakkal (S. India), diameter about 15 mm and weight about 4 g, showing the four-rayed asterism. Note that the arms of the rays do not cross at right angles.

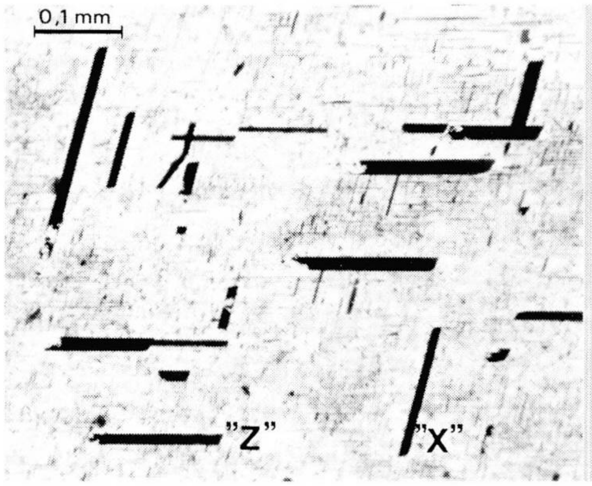

Fig. 2a

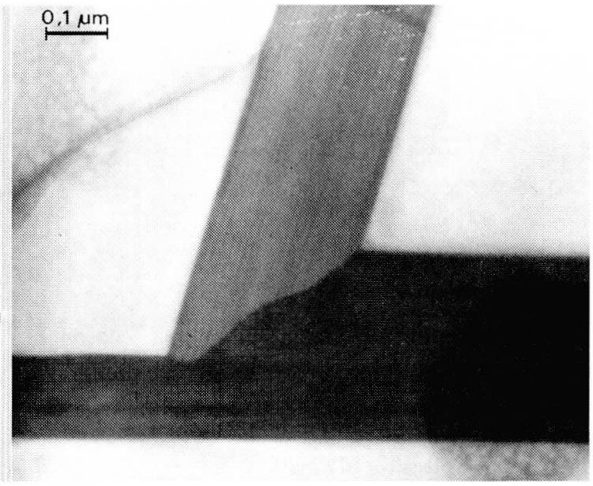

Fig. 2b

Figure 2
a. Optical micrograph of a thin section cut perpendicular to the b-axis of the diopside host. The opaque inclusions are preferentially arranged parallel to two directions, which intersect at an angle of 104°.
b. An electron microscopical bright field image of an intergrowth of the inclusions. The streaking parallel to the long axis of the inclusions is due to the Moiré effect caused by the magnetite and the underlying diopside.

viewed under the polarizing light microscope, the presence of two parallel sets of inclusions, subtending an angle of 104° (Figure 2a). An electron microscopical bright field image of the inclusions shows the relation at higher magnification (Figure 2b).

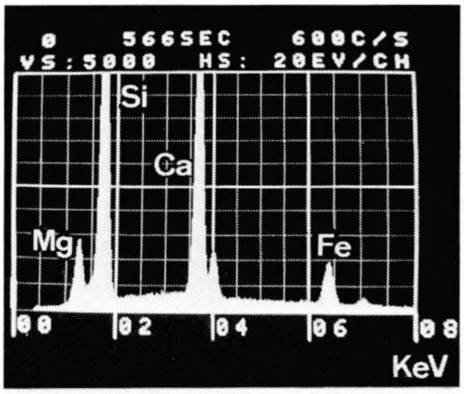

Fig. 3a

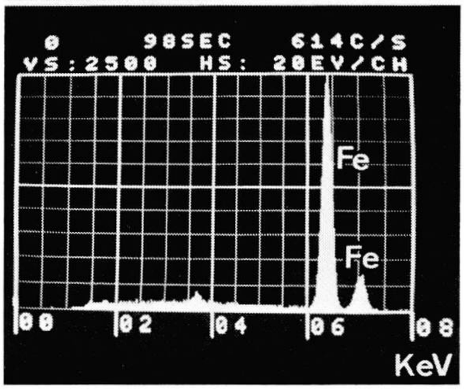

Fig. 3b

Figure 3 X-ray microanalysis of diopside (a) and a blade-shaped inclusion (b). Only Fe is present as major element in the inclusion.

Chemical Analysis

Qualitative X-ray microanalysis with a scanning electron microscope (GOLDSTEIN et al., 1981) indicates that only Fe is present in the opaque inclusions (Figure 3b). Besides Fe, only Ca, Mg an Si have been found in the diopside host (Figure 3a). According to the microprobe analysis (see Table 1), the chemical composition of the star diopside matrix is as follows $Ca_{.94}Mg_{.89}Fe^{++}_{.14}Na_{.01}Ti_{.005}Al_{.02}[Si_{1.96}Al_{.04}O_6]$.

Table 1 Chemical analysis (in weight percent) of star diopsides from Namakkal, S. India

SiO_2	53.5	54.5
TiO_2	0.2	
Al_2O_3	1.4	0.98
FeO	4.7	3.57
MgO	16.2	17.60
CaO	23.9	24.10
Na_2O	0.2	
Cr_2O_3	0.02	
Total	100.1	100.85

1. Microprobe analysis by Mr. R. Gubser
2. Microprobe analysis from Schubnel et al. (1968)

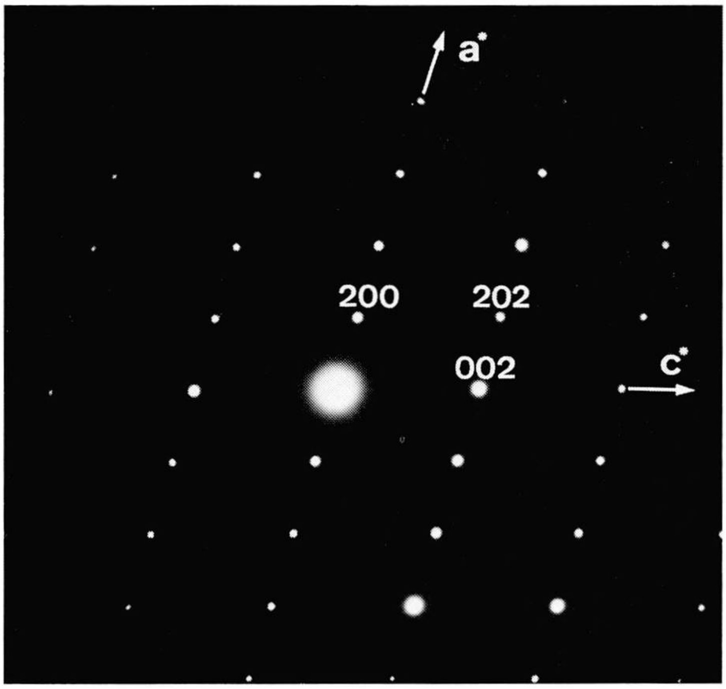

Figure 4 [010] electron diffractogram of diopside.

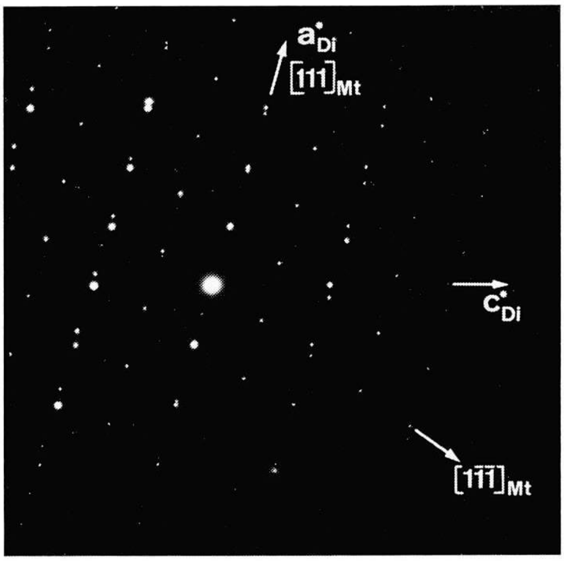

Fig. 5a

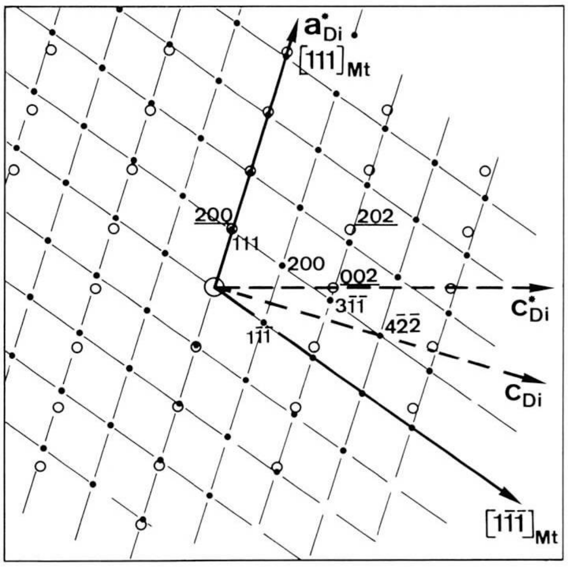

Fig. 5b

Figure 5
a. Electron diffractogram of the "Z" inclusions of magnetite (Mt) in diopside (Di).
b. Schematic diagram of the intergrowth of the "Z" inclusions. Notice characteristic relation: $\langle 111 \rangle_{Mt}$ // $[100]^*_{Di}$, while $\langle 110 \rangle_{Mt}$ // $[010]_{Di}$.

Electron microscopy

Selected area electron diffraction patterns using a transmission electron microscope have been made in order to identify the inclusions in the star diopside, and in addition to establish the crystallographic relation between the star diop-

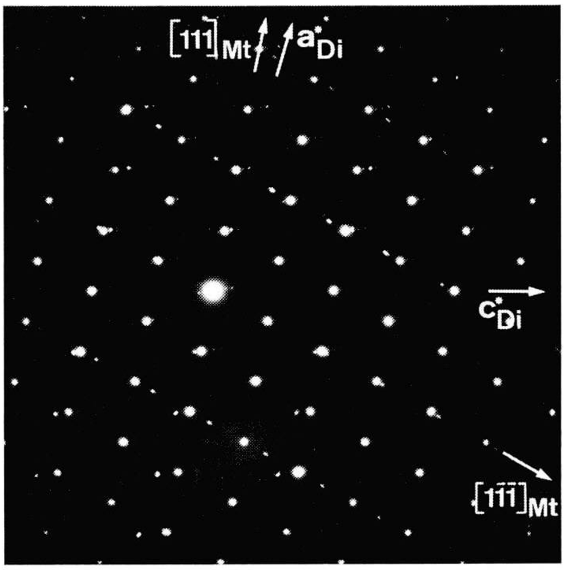

Fig. 6a

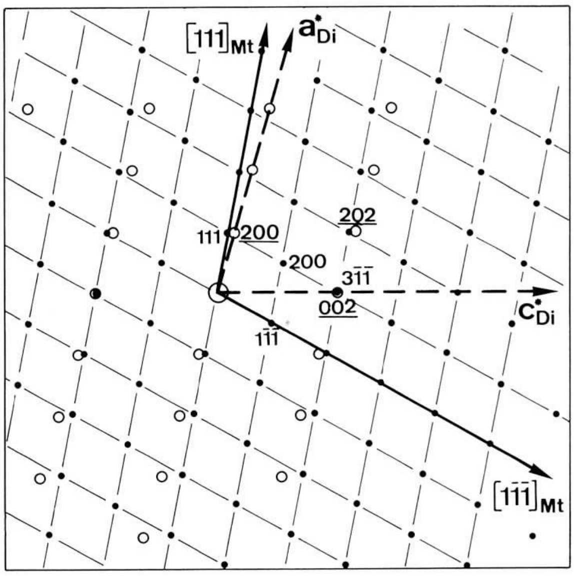

Fig. 6b

Figure 6
a. Electron diffractogram of the "X" inclusions of magnetit (Mt) in diopside (Di).
b. Schematic diagram showing intergrowth of the "X" inclusions. Here $\langle 111 \rangle_{Mt} \wedge [100]^*_{Di} = 6°$, while $\langle 110 \rangle_{Mt}$ remains parallel to $[010]_{Di}$.

side host and the inclusions. Ion-beam thinned samples have been studied with a Jeol JEM 200CX transmission electron microscope operating at 200 kV and equipped with a top-entry goniometer.

The orientation of the [010] diopside specimens is confirmed by their electron diffractograms (Figure 4). The electron diffraction patterns of the inclusions identify mostly as magnetite (Figures 5 and 6) and only sometimes as amphibole (Figure 7a).

Magnetite inclusions

The magnetite inclusions in the star diopside are always oriented in such a way as to make the ‹110› of magnetite parallel to the b-axis of diopside. Two different orientations of the magnetite blades, the "Z" and the "X" type inclusions (FLEET et al., 1980), are still possible, both parallel to (010) of diopside. They have the following characteristics:
"Z" with $[0\bar{1}1]_{Mt}$ // $[010]_{Di}$ and $[111]_{Mt}$ // $[100]^*_{Di}$ and
"X" with $[0\bar{1}1]_{Mt}$ // $[010]_{Di}$ and $[111]_{Mt} \wedge [100]^*_{Di} = 6°$.

Additional characteristics are given for the "Z" and "X" type inclusions in Table 2. The different patterns of the "Z" and "X" inclusions are given in Figures 5 and 6 respectively.

Amphibole inclusions

The amphibole lamellae are also oriented, according to the relation:
a_{Am} // a_{Di}, b_{Am} // b_{Di} and c_{Am} // c_{Di} (Figure 7a).

Table 2 Crystallographic relations between diopside host and magnetite inclusions

Type	Magnetite		Diopside	Special features	Figure
"X"	$[0\bar{1}1]$	//	$[010]$	$[111]_{Mt} \wedge [100]^*_{Di} = 6°$	6 a/b
	$[3\bar{1}\bar{1}]$	//	$[001]^*$	$[1\bar{1}\bar{1}]_{Mt} \wedge [001]^*_{Di} = 30°$	
	$[100]$	//	$[101]^*$	$(113)_{Mt}$ // $(001)_{Di}$	
"Z"	$[0\bar{1}1]$	//	$[010]$	$[111]_{Mt}$ // $[100]^*_{Di}$	5 a/b
	$[111]$	//	$[100]^*$	$[2\bar{1}\bar{1}]_{Mt}$ // $[001]_{Di}$	
				$(111)_{Mt}$ // $(100)_{Di}$	

Fig. 7a

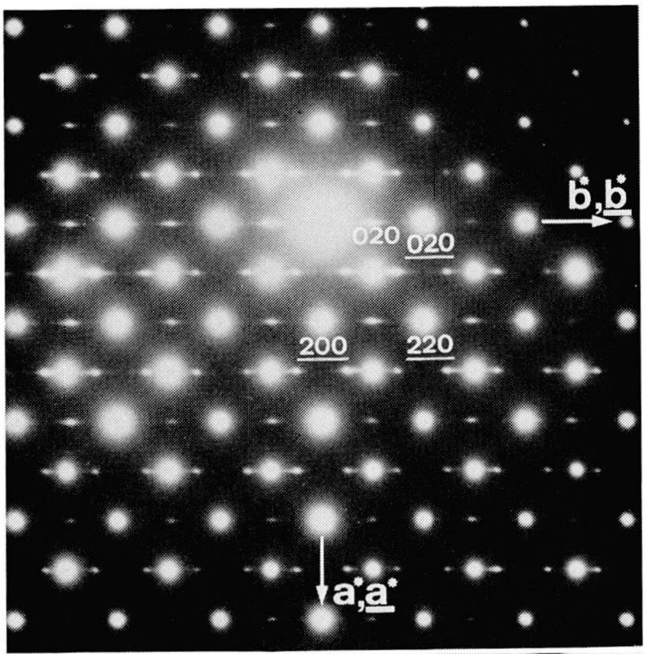

Fig. 7b

Fig. 8

Figure 7

a. [001] electron diffractogram of diopside and an amphibole inclusion. Axes and reflections of diopside are underlined.

b. Brigth field image of the amphibole inclusions. The irregular patterns are Bragg contours which do not represent any type of inclusions. An inclusions of magnetite is indicated by an arrow. The (010) lamellae of amphibole are vertical.

Figure 8 Bright field image of magnetite (Mt) and amphibole (Am) inclusions in an ion-beam thinned sample of diopsid oriented parallel to the b-axis of diopside. The "Z" type orientation of the magnetite is cut almost perpendicular to the plane of the figure.

An ion-thinned section perpendicular to the oriented "Z" type magnetite inclusions shows that these are preferentially nucleated on the amphibole lamellae (Figures 7b and 8).

Discussion and Conclusion

Intergrowths of pyroxenes and spinel in terrestrial and lunar pyroxenes have been studied by OKAMURA et al. (1976). They described two different types of intergrowths. The type I orientations are identical to our "X", and the type II orientations, correspond to our "Z". The orientation of magnetite inclusions in pyroxenes from the Grenville province has been described by FLEET et al. (1980). These authors distinguished between "X" and "Z" inclusions, which are identical to the orientations described in the present study. The "Z" orientations are almost parallel to the c-axis of the diopside host, while the "X" inclusions are slightly inclined to the a-axis of diopside. Two epitactical relations exist, one between (113) of magnetite and (001) of diopside for the "X" type inclusions, and another between (111) of the "Z" type magnetite and (100) of diopside. These two epitactical relations share as common axes ‹110› of magnetite and [010] of diopside. There is no justification in defining a three-dimensional pseudopyroxene unit cell as pointed out by OKAMURA et al. (1976) in order to explain the mechanism of magnetite exsolution. The pseudopyroxene unit cell principle would produce magnetite inclusions having both orientations present in one and the same crystal. Magnetite exsolves two-dimensionally according

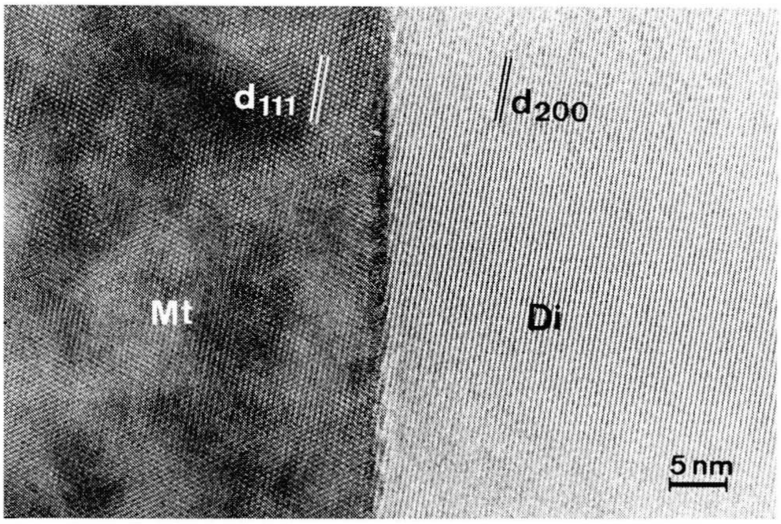

Figure 9 Detail of the intergrowth of a magnetite inclusion and the diopside host. The lattice fringes d_{200} of diopside are parallel to the d_{111} fringes of magnetite, which is characteristic for the "Z" type inclusions. The magnetite blades are bounded by irrational planes.

to two different epitactical relations as described above and shown in Figures 2, 5 and 6.

High magnification bright field images reveal that the magnetite blades are not bounded by crystal faces with rational indices. This is illustrated in Figure 9, in which the interplanar spacings d_{200} of diopside and d_{111} of magnetite are coherent, which is typical for the "Z" type inclusions. The "Z" type inclusions are inclined to the c-axis of diopside, which is parallel to the lattice fringes of d_{200} in Figure 9. The inclination angle is around 10°, while the angle "Z" $\wedge$ "X" is about 104°, which is equal to the angle subtended by the rays of the asteriated cross (Figure 1). Therefore one may conclude that the four-fold asterism of star diopside must be produced by the "Z" and "X" type inclusions of magnetite. The amphibole inclusions are of minor importance for the asterism, which is not affected by their presence.

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