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OPTICAL ANOMALY GARNET AND ITS STABILITY FIELD AT HIGH PRESSURES AND TEMPERATURES

by

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(with 4 tables, 2 figures and 1 plate)

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Abstract

The natural anisotropic garnets from the skarn deposits in Japan were found a wide range of chemical compositions of grossular-andradite solid solution. No relationship between chemical compositions and the optical properties was found.

Anisotropic garnet and its stability field have been studied in the pressure range of 250 – 1,500 bar and the temperature range of 550 – 870°C with or without H_2O and CO_2. The transition boundary from anisotropic to isotropic form was negative, and the stability field of anisotropic garnet is relatively low pressures and temperatures under H_2O and CO_2 conditions.

Introduction

Garnet comprises an important and widespread group of rock-forming minerals distinguished by their chemical diversity, close structural similarity, physical properties, and petrogenetic implications. They are cubic system which have space group *Ia3d*. Natural garnet crystals, however, often exhibit double refractions. In particular the grossular-andradite series from skarn deposits commonly show considerable birefringence. Observation of the optical character of garnets from natural samples have been the subject of voluminous literatures since the 19th Century.

Descloizeaux (1867), Rosenbusch (1873), Mallard (1876), Klein (1883), Levy (1888), etc. studied anisotropic garnets by means of optical microscopy, and discussed the origin in terms of twinning among growth sectors. Rosenbusch and Wulfing (1927) concluded that the garnets showing optical anomalies are triclinic crystals which are pseudo-cubic. Lacroix (1892) reported that optical anomalies in garnets are observed only in Ca-rich garnets, and Brogger (1890) described that the garnets in contact metamorphosed rock type show primary optical anomalies. On the other hand, Brauns (1891) and Rinne (1925) suggested that these phenomena are due to strain during the crystal growth.

Anisotropism of garnet is more marked in the grandite series, weak birefringence is rather characteristic of andradite and grossular series. Often appearing in these garnets as sector twins composed of six pyramids with vertical meeting at the center of the crystal. Anisotropic garnets also showed distinct zoning and twinning.

Recently studies with electron microprobe have made remarkable advances and new facts are gradually being elucidated. Verkaeren (1971) showed that a chemical Fe^{3+}/Al zonation corresponds to the lamellae anomaly and proposed a genetic interpretation whereby the anisotropy is related to strain induced in the crystals by the oscillation of the physico-chemical conditions during their growth. Lessing and Standish (1973) investigated an anisotropic andradite showing oscillatory zoning and dodecahedral twinning, and suggested that the zoning may be explained by rapid changes in the chemical composition of hydrothermal solutions or the oxidation state of iron, and that the observed anisotropism may be due to the substitution of ferric iron and aluminum. On the other hand, Chase and Lefever (1960) showed from the asterism of the Laue spots that birefringence of synthetic garnet is due to strain from variations in such properties as compositions, purity and perfection of the crystals, or variations in growth conditions, rather than twinning.

Takeuchi and Haga (1976) reported that the crystal of Munam garnet has a non-cubic structure based on cation ordering in the octahedral position, thereby explaining its optical anomaly. The anomaly in such a case would probably be due to more distortion of the cubic structure; the extence dodecahedral about x cations (x = Ca, Mn, Fe, or Mg) in the structure is presumably favorable with distortion.

Although a vast amount of work has been done as to the reason for the cause of optical anomalies of garnets, as well as synthetic garnet, it seems that we have not solved the problem yet. In this paper, chemical and optical properties of natural anisotropic garnets are presented and the stability field of anisotropic garnet is determined experimentally at high pressures and temperatures with H_2O and CO_2.

Occurrence of Natural Anisotropic Garnets

Yokozuru mine, Fukuoka Prefecture, is a contact metasomatic deposit in limestone by granodiorite. Garnets are associated with clinozoisite, wollastonite, hedenbergite, epidote and calcite. Anisotropism of garnets in this mine are not distinct and zonal structure is found. They are brown in color with a small crystals.

Yanagigaura mine, Fukuoka Prefecture, is also a thermal metasomatic deposit in non-metamorphic Palaeozoic formation. The mineral assemblage of this mine is garnet, magnetite, hedenbergite, epidote, ferrohastingsite, calcite, pyrite and chalcopyrite. Garnets are black brown in color and are about 0.5 to 1 cm in diameter. Anisotropic garnets of this mine have zonal structure in the rim. Sometimes the core shows sector zoning.

Kamaishi mine, Iwate Prefecture, is one of the main iron-copper deposits in Japan, the skarn of which shows a zonal arrangement. This mine is a thermal metasomatic deposit in limestone by porphyrite and/or granodiorite. Garnets occur commonly in skarn and iron ore deposits, but anisotropic garnets occur only in skarn. They have usualy zonal structures and sector zoning and associated with epidote, actinolite, hedenbergite, calcite and quartz. Garnets are brown to red brown in color and are about 0.5 to 1.5 cm in diameter.

Kamioka mine, Gifu Prefecture, is composed of pyrometasomatic skarn and hydrothermal deposit. The skarn in this mine are genetically divided into the following three groups; 1, recrystallized skarn, formed by a regional metamorphism of impure limestone, 2, zoned skarn along the contact between limestone and syenitic rock, 3, pyrometasomatic skarn, formed by pyrometasomatic replacement of limestone. Garnets are usualy yellow to yellow brown in color and are about 1 to 3 mm in diameter. Garnets occur with hedenbergite, calcite and quartz. Anisotropic garnets of this mine have zonal structure and sometimes sector zoning. Fig. 1 in Plate showns sector zoning and its optical orientation of one of the samples from this mine.

Tsumo mine, Shimane Prefecture, is a thermal metasomatic deposit in limestone by granite porphyry. The garnets are very distinct in optical anomaly, they are brown to deep green in color, however, anisotropic garnet is usualy brown in color and shows zonal structure. Garnets associated with vesuvianite, epidote, scheelite, fluorite, hedenbergite and quartz.

Chemical and Physical Properties of Natural Anisotropic Garnets

Optical data for the anisotropic garnets from some localities are given in Table 1. The optical properties of each of the crystals are slightly different. The index of refraction for the isotropic part of garnet crystal from Yanagigaura mine is n = 1.886, and for the anisotropic part is slightly low, n = 1.864. Kennedy (1953) reported that garnets from Jambo Basin, Alaska are twinned, with 2V approximatly 90° and with birefringence, estimated at 0.005. Sample number of 175–2 from Tsumo mine has notable birefringence, estimated 0.010. The optical axial angles of natural anisotropic garnets were sometimes positive and sometimes negative with different angles as also shown in Table 1.

Table 1. Optical properties of natural anisotropic garnets

Sample No.	Locality	Refractive indices	Cell parameter (Å)	Composition**	2V
36A	Yokozuru	1.880	12.045 ± 0.004	$Gro_8 And_{92}$	n.d.
21A	Yanagigaura	1.864	12.039 ± 0.001	$Gro_{11} And_{89}$	+74 ~ +84
K1	Kamaishi	n.d.	11.960 ± 0.004		n.d.
5-13	Kamaishi	n.d.	11.937 ± 0.003		+68 ~ +78
6-8	Kamaishi	1.818 ± 0.006	11.970 ± 0.01	$Gro_{46} And_{54}$	n.d.
11-10	Kamaishi	1.792 ± 0.005	11.913 ± 0.002	$Gro_{68} And_{32}$	n.d.
1209	Kamioka	1.791	11.924 ± 0.005	$Gro_{68} And_{32}$	+68 ~ +82
175-2	Tsumo	1.758 ± 0.002*	11.884 ± 0.001	$Gro_{84} And_{16}$	−78 ~ −86
50-4	Tsumo	1.884 ± 0.002	12.062 ± 0.001	$Gro_6 And_{94}$	n.d.

* $\alpha = 1.757, \beta = 1.761, \gamma = 1.767, \gamma\text{-}\alpha = 0.010$
** estimated values from index and cell parameter

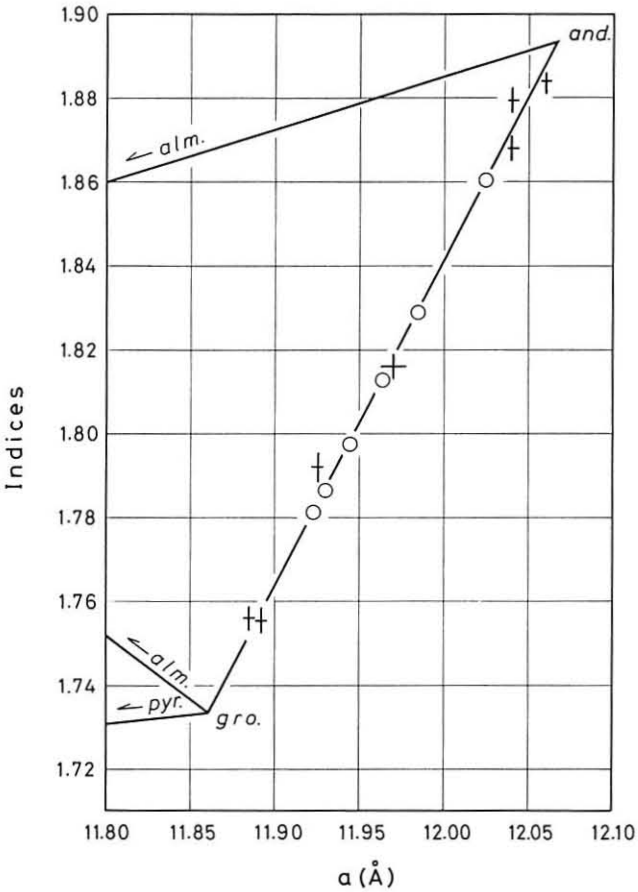

Fig. 1. Relationship between refractive indices and cell-parameters of natural (cross) and synthetic (open circle) optical anomaly garnets.
and; andradite, gro; grossular, pyr; pyrope, alm; almandine

Table 2 Chemical composition of garnet from Yokozuru

	isometric part	anisotropic part
SiO_2	38.70	38.79
TiO_2	0.69	0.50
Al_2O_3	13.95	14.40
Fe_2O_3	11.42	11.31
MnO	0.25	0.22
MgO	0.35	0.35
CaO	34.88	34.93
Na_2O	0.01	0.00
K_2O	0.01	0.00
Sum	100.26	100.50
Number of cations on a basis of 24 oxygens		
Si	6.027	6.019
Al^{IV}	0.000	0.000
Al^{VI}	2.561	2.633
Ti	0.080	0.058
Fe^{3+}	1.338	1.320
Mn	0.033	0.028
Mg	0.082	0.082
Ca	5.820	5.806
Na	0.004	0.000
K	0.001	0.000
End-member molecules per cent		
Grossular	59.90	61.70
Andradite	38.62	37.09
Spessartine	0.49	0.49
Pyrope	1.21	1.21

$(Ca_{2.90}Mg_{0.04}Mn_{0.01})_{2.95}(Al_{1.32}Fe^{3+}_{0.66}Ti_{0.03})_{2.01}$
$Si_{3.01}O_{12.00}$ for anisotropic part
$(Ca_{2.91}Mg_{0.04}Mn_{0.02})_{2.97}(Al_{1.28}Fe^{3+}_{0.67}Ti_{0.04})_{1.99}$
$Si_{3.01}O_{12.00}$ for isometric part

Relationship between refractive indices and cell-parameters of anisotropic garnets is shown in Fig. 1. The chemical compositions of natural anisotropic garnets show a wide range of grossular-andradite solid solution. The end members do not show optical anomaly. The compositional variation of zoning patterns in isotropic and anisotropic parts from Yanagigaura mine were analyzed by electron microprobe. From band to band in zoning patterns, when Al decreases Fe increases. The selected partial analyses of the isotropic and anisotropic parts in garnet from Yokozuru mine are listed in Table 2. The compositional variation among these two parts is not so high. MgO and MnO contents are usually less than 0.35 wt%. These garnets are typical solid solution of grossular and andradite.

In previous works, several chemical analyses have been made on the chemical differences between the isotropic and the anisotropic garnets. Verkaeren (1971) reported that isotropic band are Fe-rich and Al-poor compared with the anisotropic band in zonal structure of garnet, whereas Lessing and Standish (1973) found the opposite tendency, i.e. the isotropic band are Al rich. Murad (1976) discussed the relationship between chemical composition and birefringent zones of garnet from the Thera Island by analyses of electron microprobe. He found that the birefringent zones had high Al concentrations and isotropic zones were almost pure andradite which is an extreme depletion of Al. On the other hand, Kano and Yashima (1976) found anisotropic bands in almandine rich garnets from rhyolite and dacite. These bands are slightly richer in Ca, Mn and Ti than isotropic parts. These data show no agreement as to the chemical differences between the two types of garnets.

From the previous works and recent studies of chemical analyses in both the isotropic and the anisotropic garnets, no relationship between chemical composition and optical anomaly of garnets was found. However, garnets without Ca component do not show birefringence. These results show that character such as optical anomaly of garnet does not depend upon variation of chemical composition but also distortion of crystal structure.

High Temperature Experiment on Anisotropic Garnet

High temperature experimental work by Merwin (1915) shows birefringent types of garnet which have composition about $\mathrm{grossular}_{25}\mathrm{andradite}_{75}$ solid solution inverted at about 800°C to a strictly isotropic form. A similar experiment was performed by Stose and Glass (1938) on a sample of the anisotropic andradite. At 860°C the anisotropic grains became isotropic, and did not invert on cooling down. Kozu et al. (1940) also reported that optical anomaly garnet from the Kamaishi mine inverted to a isotropic form at 1000°C. Ingerson and Barksdale (1943) observed that in an iridescent garnet from Nevada the birefringence did not decrease untill the temperature reached 1060°C and continued almost to the melting point. Also Allen and Fahey (1957) observed that the andradite from the Pewabic mine lost their birefringence at 860°C.

Thermal experiments under atmospheric pressure have been made on the samples of anisotropic garnets. All the samples were crushed into the coarse powder. Experimental results are shown in Table 3. Anisotropic garnets invert to isotropic form at 850 to 955°C. The inverted temperatures between sample

Table 3 Experimental results of heating and annealing for anisotropic garnets

Sample No.	Heating conditions		Results after heating	Annealing conditions		Results of annealing
	Temp. (°C)	Time (hr.)		Temp. (°C)	Time (hr.)	
Yokozuru 36A						
Gro_8And_{92}						
Yo1	945+15	44.5	isometric	800	72	anisotropic
Yo2	〃	〃	〃	600	41.5	〃
Yanagigaura 21A						
$Gro_{11}And_{89}$						
Ya1	855+15	43	〃	800	72	isometric
Ya2	〃	〃	〃	600	41.5	anisotropic
Ya3	900	13.5	〃	200	5	〃
Ya4	〃	〃	〃	100	652.5	〃
Ya5	〃	14	〃	600	11	〃
Ya6	〃	88.5	〃	600	72	isometric
Ya7	1050	66	〃	600	95	〃
Kamaishi						
$Gro_{58}And_{42}$	885+15	65	〃			
Tsumo 175-2						
$Gro_{84}And_{16}$						
Ts1	945+15	44.5	〃	800	72	anisotropic
Ts2	〃	〃	〃	600	41.5	〃

Gro; grossular, And; andradite.

numbers 36A and 21A which have almost the same compositions were observed to have large difference of the inverted temperatures probably depends on the order-disorder of crystal structure rather than the composition of anisotropic garnets. Inversion from anisotropic to isotropic form is reversible. For instance, in sample numbers of 36A and 175–2 optical anomaly appeared again by annealing at 800 and 600°C after heating at around 900°C. But annealing run at 800°C on the sample 21A, optical anomaly did not appear, it appeared again at 600°C. Heating samples at higher temperatures did not appear optical anomaly by annealing. These phenomena may be explained by the anisotropic garnet inverted to complete disorder form by heating, this garnet does not appear optical anomaly by annealing.

The temperature to which birefringent varieties of grossular-andradite solid solution have to be heated for them to loss their birefringence has been used as an indication of the temperature of their crystallization. Murad (1976) concluded from his experiment that although cyclic variations in the composition of circulating fluids could have brought about the zoning, this may also be the result of temperature fluctionations, individual garnet compositions being indicative of the respective formation temperature.

Synthesis of Anisotropic Garnet and its Stability Field

Anisotropism is more marked in the grandite series in garnet solid solutions, especially in the grossular-andradite series. We have recently synthesized anisotropic garnet of grossular-andradite solid solution at hydrothermal conditions. Anisotropic garnet forms at 650°C and 800 bar from a oxide and carbonate mixture for starting material, with more than 10 wt% water. This garnet has beautiful anomaly with sector growth (Fig. 2 in Plate). Synthetic anomaly garnets did not show zonal patterns in any experimental runs. The synthetic crystal of this anisotropic garnet is colorless, and 30μ in diameter.

To investigate the stability field of pressures and temperatures on the

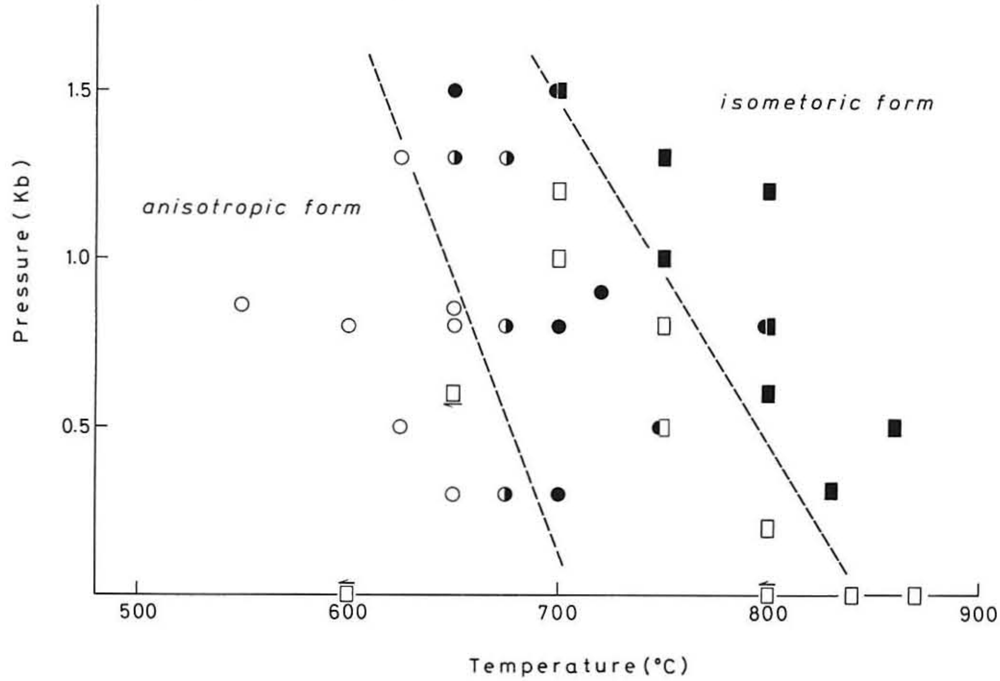

Fig. 2. Stability field of natural and synthetic optical anomaly garnets.
open circle: synthesized anisotropic garnets. closed circle: synthesized isotropic garnets.
open rectangle: natural anisotropic garnets. closed rectangle: natural isotropic garnets.

formation of anisotropic garnet, natural sample (21A) and oxide, carbonate mixture were used for starting materials. As the starting material mixtures of the compositions between grossular and andradite were prepared from $CaCO_3$, Al_2O_3, Fe_2O_3 and SiO_2 with stoichiometric proportions. All runs were made by test tube type hydrothermal apparatus with and without H_2O and CO_2. It was difficult to determine inversion from anisotropic to isotropic form with the natural sample below 700°C, since this inversion rate is extremely slugish at low temperature.

It is very important that both of H_2O and CO_2 influences the forming of anisotropic garnet. Because, in nature anisotropic garnet occurs mainly in contact metasomatic area in limestone by igneous rocks. For the investigation of the effect both of H_2O and CO_2, we used crystalline phase assemble which was heated mixture of oxide and carbonate at 1100°C for 20 hr, and used $Ca(OH)_2$ insted of $CaCO_3$ for starting materials. All experimental results are shown in Table 4 and Fig. 2.

Anisotropic garnet forms at 650°C and 800 bar from an oxide and carbonate mixtures with more than 10 wt% water. However, anisotropic garnet did not form at the same pressure-temperature condition with 5 wt% water. With starting material of $Ca(OH)_2$ plus oxide mixture with 10, 30 and 80 wt% of water, no anisotropic garnet was formed. In a run of the same starting material with CO_2 buffer (X_{CO_2} = 0.05), anisotropic garnet is clearly synthesized. According to these experimental results, we pointed out that anisotropic garnet occurs under CO_2 pressure with excess water in low pressures and temperatures.

Kalinin (1976) and Shoji (personal communication 1975) reported that synthesized garnets which compositions were grossular and andradite end-members never showed optical anomaly. We also synthesized garnets of andradite end-member and grossular$_5$-andradite$_{95}$ solid solution with H_2O and CO_2. These garnets showed isotropic forms.

Stability fields of natural anisotropic garnet and synthetic garnet are shown in Fig. 2. Pressure-temperature curves of inversion from anisotropic to isotropic forms for natural and synthetic samples are negative. As clearly recognize from Fig. 2, stability field of anisotropic grossular$_{50}$-andradite$_{50}$ solid solution which was synthesized from oxide-carbonate mixture shows a slightly different area than that of natural anisotropic garnet. Stability curve of the synthetic sample is lower than the natural sample. This phenomenon may be explained by that chemical composition or degree of order-disorder between synthetic and natural garnets are different.

Table 4 Experimental results at high pressure and temperature runs

Starting materials	Pres. (bar)	Temp. (°C)	Time (days)	H_2O or CO_2	Results
Natural anisotropic garnet 21A					
	1500	700	37	no	isotropic
	1200	〃	34	〃	anisotropic
	1000	〃	20	〃	〃
	1300	750	11	〃	isotropic
	1000	〃	12	〃	〃
	800	〃	7	〃	anisotropic
	500	〃	〃	〃	〃
	1200	800	〃	〃	isotropic
	800	〃	12	〃	〃
	600	〃	16	〃	〃
	200	〃	7	〃	anisotropic
	300	830	11	〃	isotropic
	500	860	16	〃	〃
Oxide and carbonate mixtures					
$Gro_{70}And_{30}$	800	600	25	yes	anisotropic
	850	650	25	〃	〃
	800	700	25	〃	optical anomaly not clear
$Gro_{50}And_{50}$	850	550	25	〃	anisotropic
	800	600	〃	〃	〃
	〃	650	37	〃	〃
	850	〃	55	〃	〃
	1300	〃	42	〃	optical anomaly not clear
	800	675	28	〃	〃
	300	700	〃	〃	isotropic
	800	〃	31	〃	optical anomaly not clear
	1500	〃	36	〃	isotropic
	500	750	42	〃	〃
	800	650	27	H_2O 40wt%	anisotropic
	〃	〃	〃	H_2O 10wt%	〃
	〃	〃	31	H_2O 5wt%	isotropic
$Gro_{30}And_{70}$	〃	600	25	yes	anisotropic
	850	650	55	〃	〃
	800	700	25	〃	〃
Gro_5And_{95}	1300	675	21	〃	isotropic
	800	650	〃	〃	〃
And_{100}	〃	〃	〃	〃	〃

Starting materials	Pres. (bar)	Temp. (°C)	Time (days)	H_2O or CO_2	Results
Crystalline phases					
$Gro_{70}And_{30}$	500	500	30	X_{CO_2}=0.25	anisotropic
	800	〃	〃	no	?
$Cro_{50}And_{50}$	500	〃	〃	X_{CO_2}=0.25	anisotropic
	800	600	〃	no	?
$Cro_{30}And_{70}$	500	500	〃	X_{CO_2}=0.25	anisotropic
	800	700	〃	no	?
Oxide+Ca $(OH)_2$ mixture					
$Gro_{40}And_{60}$	800	650	31	H_2O 10wt%	isotropic
	〃	〃	33	H_2O 30wt%	〃
	〃	〃	〃	H_2O 80wt%	〃
	〃	〃	27	X_{CO_2}=0.05	anisotropic

Gro; grossular molecule, And; andradite molecule.

Discussion

Although a vast amount of works have been done in the field of the causes for anisotropic garnet, it seem that we have not yet solved this problem. Birefringence of garnet appears in intermediate series of grossular-andradite solid solution. Optical anomalies will develop only in Ca-garnets and contain some amount of other components of grossular-andradite series, in which the only major chemical variation is expected in the amount of Al_2O_3 and Fe_2O_3, which change inversely. An alternative explanation for the development of the zoning is provided by the results of experimental work by Huckenholz et al. (1974) who found that, at constant pressure and bulk chemical composition, the grossular content of granditic garnets synthesized from fassaite-bearing assemblages tends to increase with falling temperatures. Applied to the garnet from Thermia Island by Murad (1976), this would indicate higher formation temperatures for the isotropic parts and birefringent zone with the high grossular/andradite ratio indicates the formation of these to have come to a gradual decreasing temperature.

However, effect of H_2O and CO_2 pressures are also important conditions for the formation of anisotropic garnet. The oscillation zoning of the anisotropic and isotropic garnets may form under physical and chemical environments, not only cyclic variations of temperature, but chemical conditions with H_2O and CO_2. Chase and Lefever (1960) showed that birefringence of garnet is due to strain from variations in such properties as composition, purity and perfection of the crystal, or variation in growth conditions. However, in recent experiments no evidence was found to suggest strain among the crystal growth at hydrothermal conditions. A possibility of an order-disorder relationship, as in the feldspars, may also be considered.

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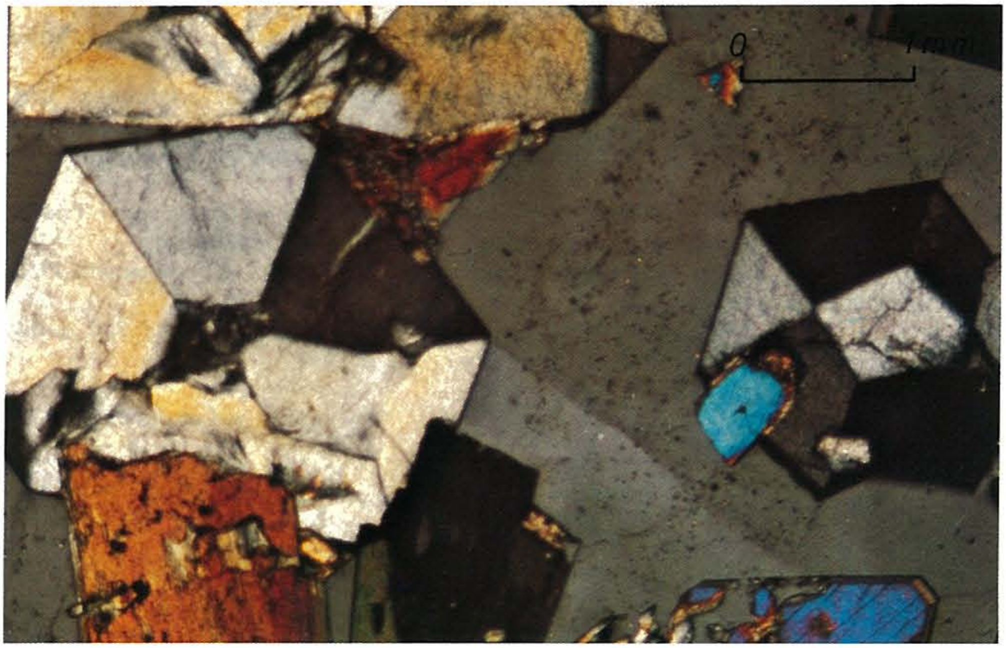
Explanation of Plate

Fig. 1 Sector zoning of natural anisotropic garnet from Kamioka mine. (crossed nicols).

Fig. 2 Sector zoning of synthetic optical anomaly garnet at 650°C and 800 bar hydrothermal condition (crossed nicols plus gypsum plate).

Plate

1



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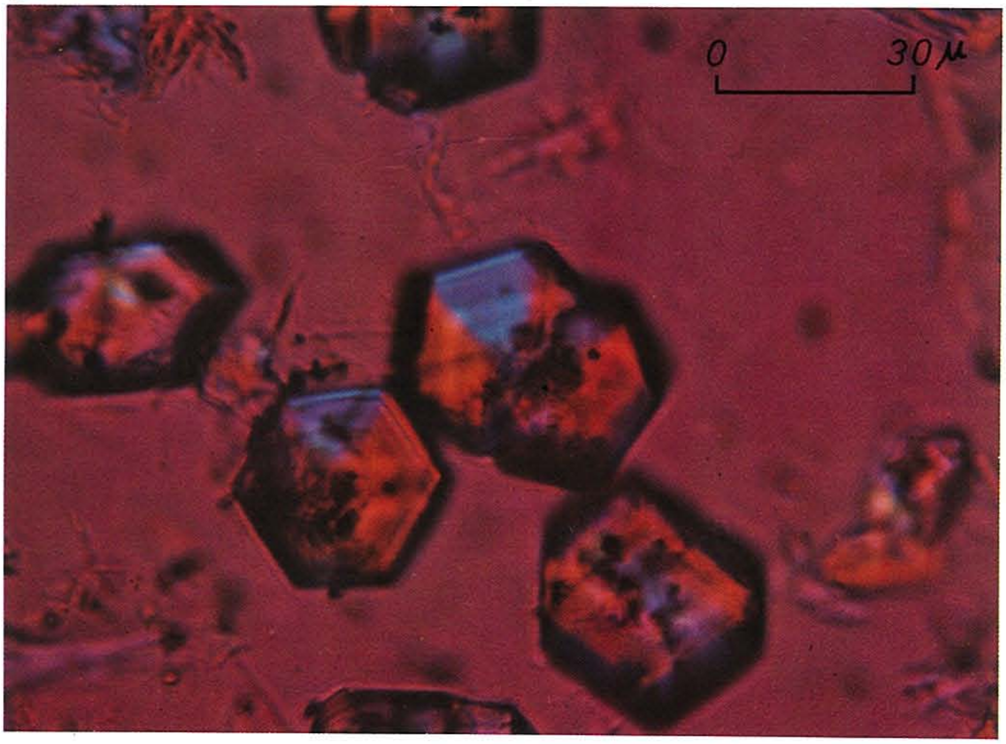

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