

## XIV. MINERALOGY AND INTERNAL STRUCTURE OF MANGANESE NODULES OF THE GH80-5 AREA

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### Introduction

Mineral composition and internal structure of manganese nodules are most important in determining their physical and chemical characteristics. Earlier investigations have shown that surface features (rough to smooth) of nodules are almost exclusively correlated to constituent minerals, and that similar features corresponding to rough and smooth surfaces are also present inside nodules (USUI, 1979). Therefore precise mineralogical description gives us more detailed external and internal characteristics of nodules than simple morphological description.

In order to confirm the relationships of mineral composition to external and internal morphology and chemical composition, powdered samples were prepared from defined portions of each nodule. Comparative studies were made on the same samples by means of X-ray powder diffraction method, microscopy and chemical analyses.

Internal structure of the samples are described on the basis of megascopic and microscopic observations on polished sections taking the X-ray diffraction data into account. One or more nodule samples were selected from each station mainly along detailed survey lines (USUI, Chapter IX in this cruise report).

### Determination of mineral constituents

Mineralogy of marine manganese minerals has been controversial because of their low crystallinity and small crystal size. Terminological problems contended by many workers are summarized by BURNS and BURNS (1977, 1979). Table XIV-1 lists reported mineral terms of marine manganese nodules. I adopt following terms in this article; 10 Å manganate, 7 Å manganate, and  $\delta$ -MnO<sub>2</sub>. The terms  $\delta$ -MnO<sub>2</sub> is restricted to the 2 diffraction line type.

These mineral constituents can be determined by X-ray diffraction method and reflecting microscope independently. The X-ray diffraction method gives semi-

Table XIV-1 Comparison of mineral terms for marine manganese nodules

10 Å manganite	7 Å manganite	$\delta$ -MnO <sub>2</sub>	Buser and Grütter (1956)
todorokite	birnessite	$\delta$ -MnO <sub>2</sub>	Burns and Burns (1977)
buserite	birnessite	$\delta$ -MnO <sub>2</sub> (random-stacked or tubostratic)	Giovanoli and Burki (1975)
todorokite	birnessite	vernadite	Chukhrov et al. (1979)
10 Å manganate	7 Å manganate	$\delta$ -MnO <sub>2</sub> (Z-disordered)	Arrhenius et al (1979)
10 Å manganate	7 Å manganate	$\delta$ -MnO <sub>2</sub>	This study

Table XIV-2 Criteria for optical determination of nodule minerals under reflecting microscope (Usui, 1979)

	10 Å manganate phase	$\delta$ -MnO <sub>2</sub> phase
Color	light grey	dark grey
Reflectivity (wave length 460 nm)	high (ca. 13%)	low (ca. 8%)
Anisotropism	strong	none
Hardness (Vicker's hardness number)	high (55-112, mean 82)	low (10-24, mean 17)
Internal reflection	none	none

quantitative mineral contents in nodules while the microscopy precise microstructure of the mineral constituents in each nodule sample.

X-ray powder diffraction analyses were accomplished in the constant measurement conditions using the diffractometer Type RAD-rA (Rigaku Denki Co. Ltd.) with a monochromator. Powder samples mounted in standard glass holders were subjected to nickel-filtered CuK $\alpha$  radiation at 40 kV and 160 mA during 8° (2 $\theta$ )/min scan from 3° to 45° (2 $\theta$ ). Manganese minerals were identified on diffractograms according to six characteristic peaks at 10, 7, 5, 3.5, 2.4 and 1.4 Å d-spacings. 10 and 5 Å reflections are diagnostic of 10 Å manganate, while 2.4 Å reflections is responsible to both 10 Å manganate and  $\delta$ -MnO<sub>2</sub>. Amounts of 10 Å manganate are estimated from the peak height at 10 Å. Amounts of  $\delta$ -MnO<sub>2</sub> are estimated from the peak heights at 10 and 2.4 Å assuming that the intensity ratio at 10 and 2.4 Å reflections of 10 Å manganate is 5.0. Analytical error of the amounts is in the order of 10%. Accessory silicate minerals, such as quartz, feldspars, clay minerals and zeolites, are examined on the basis of ASTM data files.

Microscopic identification of manganese minerals is based on earlier works. Two principal manganese phases, the 10 Å manganate and  $\delta$ -MnO<sub>2</sub>, are distinguishable on reflecting microscope according to reflectivity and anisotropism, except for sub-microscopic lamination of both phases. Silicate minerals are hard to identify under reflecting microscope. The criteria for microscopic identification of manganese minerals are listed in Table XIV-2.

### X-ray diffraction analysis

The X-ray diffraction study has shown that dominant manganese minerals of these nodules are 10 Å manganate and 2 line form  $\delta$ -MnO<sub>2</sub> but distinct 7 Å reflections responsible to manganese minerals are not found, which is consistent with our earlier work (USUI, 1982). Other manganese-free minerals in manganese nodules determined are quartz, plagioclase, and montmorillonite. The results of X-ray diffraction analyses are listed in Table XIV-3. The analysis numbers in the table are comparable to those for chemical analyses in Table XV-1.

To examine the relationship between surface structure and mineral composition, powder samples were taken from defined portions of nodules. The types of surface feature described for GH80-5 nodules, s, s·r, r, are variably developed on nodule tops, bottoms, and entire surfaces (USUI, Chapter IX in this cruise report). The results of these X-ray diffraction analyses reveal that the surface feature is exclusively correspondent to either of the two constituent minerals. Smooth surface (s) is always

Table XIV-3 Relative mineral composition of manganese nodules

Area I											
Line	AN*	St./Sample No	Nodule sample			Mineral abundance					
			Shape	Surface	Part	TM	DM	Qtz	Ph	Pc	Mmt
1	1979	FG252-1	IS	1	O (1-3)	tr	××	×	—	—	—
2					I	tr	××	×	××	—	—
3	1983	FG256-1	D	3	W	×	×	××	×	tr	—
4		FG256-2	D	3	T (0-2)	×	××	××	—	—	—
5					B (0-1)	×××	tr	××	—	—	tr
6	1984	B34	S	6	W (0-7)	××	—	×××	tr	—	×
7		FG257	S	6	W (0-7)	×××	—	××	××	—	—
8	1985	FG258-1	S	6	W (0-10)	××	tr	××	×	—	tr
9	1988	FG261-1	S	1	O (0-3)	tr	×	×	—	—	—
10					I	—	××	×	××	×	—
11		FG261-2	D	1	W	tr	××	×	×	—	—
12	1989	B36	D	1	W	tr	××	××	—	—	—
13	1990	FG236-1	D	1	W	×	××	×	tr	—	—
14	1991	B37	DP	1	W	tr	××	×	×	—	—
I-a 15	1987	B35	S	6	W (0-10)	××	—	×	—	—	—
16		FG260	S	6	W (0-10)	××	tr	××	×	tr	tr
17	1992	FG267	S	6	W (0-10)	×××	×	×××	×	—	—
18	1993	FG268	S	6	W (0-10)	×××	—	×××	tr	—	—
19	1994	B38	S	6	W (0-10)	××	×	×××	tr	—	—
20	1995	FG269	S	6	W (0-10)	×××	×	××	—	tr	—
21	1996	FG270	ISP	3	T (0-1)	×	×	××	××	—	tr
22					B (0-2)	×	×	××	××	—	tr
23	1997	FG271	S	6	W (0-10)	××	×	×××	tr	—	—
24	1998	B39	S	4	W	××	tr	××	×	—	—
25			D	4	W	××	×	××	tr	—	—
26	1999	FG272	IS	2	T (0-2)	×	×	××	—	—	—
27					I	tr	××	×	××	—	—
28					B (0-1)	×	×	××	—	—	—
29	1986	P196	ID	2	T (0-2)	×	×	××	—	—	—
30		FG259-1	IDP	2	T (0-2)	tr	×	×	tr	—	—
31					I	tr	××	×	××	—	tr
32					B (0-2)	××	×	××	tr	—	tr
I-b 33	1982	FG255-2	D	2	T (0-2)	××	×	×	—	—	—
34					I	—	××	×	tr	—	—
35	2000	FG273	DP	1	T (0-2)	tr	××	×	—	—	—
36					I	—	××	×	tr	—	—
37	2001	FG274	ID	1	O (0-2)	×	×	××	—	—	—
38					I	×	××	×	tr	—	tr
39	2002	FG275	ISP	1	O (0-3)	tr	×	××	—	—	—
40					I	tr	××	×	tr	×	tr
41	2003	B40	ISP	3	T (0-2)	×	×	××	—	—	—
42					I	—	××	×	×	—	—
43					B (0-1)	×××	—	××	—	—	—
44	2004	FG276	SP	3	T (0-2)	×	×	××	—	—	—
45					I	××	×	××	××	—	—
46					B (0-1)	×	×	××	—	—	—
47	2005	FG277	S	6	W (0-5)	××	—	×××	×	—	—
48			D	6	O (0-2)	××	×	××	tr	—	—

\* : analysis number.

Table XIV-3 (continued)

Line AN	St./Sample No.	Nodule sample			Mineral abundance						
		Shape	Surface	Part	TM	DM	Qtz	Ph	Pc	Mmt	
49				I (2-10)	x	x x	x x x	x x	x x	tr	
50	2006	P199	S	6	W	x x x	—	x x x	x x	tr	—
51	2007	FG278	S	6	O (0-10)	x x x	—	x x	tr	tr	—
52					I	x x	x	x x	x x	tr	—
53	1981	B33	S	6	O (0-15)	x x x	—	x x	tr	—	tr
54					I	x x	tr	x x	x x	—	—
55		FG254	S	6	W (0-10)	x x x	—	x x	—	—	—
56	2008	FG279	S	6	W (0-15)	x x	x	x x	x x	x	—
57	2009	FG280	D	6	W (0-20)	x x x	—	x x	tr	—	—
58	2010	FG281	S	6	W (0-5)	x x x	—	x x	tr	—	—
59			D	6	W (0-20)	x x x	—	x x	tr	—	—
60	2011A	FG285	D	4	O (0-2)	x x	x	x x	—	—	tr
61					I	x x	—	x	tr	—	—
62	2012	FG282	S	6	W (0-4)	x x x	—	x x x	—	—	—
63	2013	FG283	S	6	W (0-10)	x x x	—	x x	—	—	—
64					6	W (0-20)	x x	tr	x x	tr	—
65	2014	B41	D	5	O (0-1)	x x x	—	x x	—	—	x
66					I	x	x	x x	x x	x	tr
67	2015	FG284	IDP	2	O (0-2)	x	x	x x	—	—	—
68					I	x	x	x x	x x	x	—
69	1980	FG253-1	IDP	3	T (0-2)	tr	x	x	—	—	—
70					I	x	x	x	x x	—	—
71					B (0-1)	x x x	—	x	—	—	—

## Area II

Line AN	St./Sample No.	Nodule sample			Mineral abundance						
		Shape	Surface	Part	TM	DM	Qtz	Ph	Pc	Mmt	
72	2019	B43	S	6	W	x x x	—	x x x	tr	—	—
73	2020	B44	D	6	W	x x x	—	x x x	tr	—	—
74		FG289	D	6	O (0-3)	x x x	—	x x x	tr	tr	—
75					I (3-8)	—	x x	x x x	x x	x x	x x
76	2023	EG292	I	6	W	x x	x	x x	—	x	x
77	2022	FG293	ID	6	O (0-2)	x x x	—	x x x	tr	—	—
78					I	tr	x x	x x	tr	—	—
79	2025	FG294	I	6	W	x x	x	x x x	tr	—	—
80	2028	FG297-2	IS	1	O (0-2)	x	x	x x	—	—	—
81					I	—	x x	x	—	x	x
II-a	2021	FG290-1	D	6	W	x x x	—	x x x	tr	tr	—
83		FG290-2	S	6	W	x x x	—	x x x	—	—	—
84	2041	FG304	SP	6	O (0-2)	x x	—	x x x	tr	—	—
85					I	x	x	x x	x x	—	—
86	2042	B55	S	6	W	x x x	tr	x x	—	—	—
87	2043	FG305	D	6	W	x x	tr	x x	tr	tr	tr
88	2044	B56	ID	5	O (0-1)	x x	tr	x x	—	—	—
89					I	tr	x x	x x	x x	x	tr
90	2046	FG307	IDP	5	T (0-2)	x	x x	x x	tr	—	—
91					I	x	x x	x x	x x	—	tr
92					B (0-1)	x x x	tr	x x	—	—	—

Table XIV-3 (continued)

Line	AN	St./Sample No.	Nodule sample			Mineral abundance					
			Shape	Surface	Part	TM	DM	Qtz	Ph	Pc	Mmt
93		P206	ID	4	O (0-3)	x	xx	x	—	—	—
94					I	tr	xx	xx	xx	tr	—
95	2045	FG306	IS	2	T (0-1)	x	x	xx	—	—	—
96					I	tr	xx	x	x	tr	tr
97					B (0-1)	xx	x	x	—	—	—
98	2022	B45	ID	2	T (0-3)	tr	xx	xx	—	—	—
99					I	x	xx	x	x	tr	—
100					B (0-1)	xx	x	x	—	—	—
101		FG291	ID	2	O (0-2)	x	x	x	—	—	—
102					I	—	xx	x	tr	tr	—
II-b 103	2026	FG295-2	D	6	O (0-2)	xx	tr	xx	—	—	—
104					I	x	x	xx	xx	tr	tr
105		FG295-1	S	6	O (0-2)	xx	tr	xx	x	—	—
106					I (2-10)	x	x	x	xx	—	tr
107	2034	B51	D	6	O (0-1)	xxx	—	xx	—	—	—
108					I	xx	tr	xx	tr	tr	—
109	2033	FG300	S	6	O (0-10)	xxx	—	xxx	—	—	—
110					X (10-20)	xxx	—	xxx	tr	—	—
111					I (20-40)	x	xx	xx	tr	x	—
112	2032	B50	SP	6	O (0-1)	xxx	x	xx	—	—	—
113					X (1-10)	xx	tr	xx	tr	—	—
114					I (10-40)	—	xx	xx	tr	—	—
115	2031	FG299	IS	2	O (0-3)	xx	tr	xx	—	—	—
116					I	—	xx	xx	x	x	tr
117	2030	B49	ID	2	B (0-1)	xxx	—	xx	—	—	tr
118					T	x	xx	xxx	—	—	—
119					I	tr	xx	x	—	—	—
120	2029	FG298	ID	2	T (0-2)	x	xx	xx	—	—	—
121					I	—	xx	x	—	—	—
122					B (0-1)	x	x	xx	—	—	—
123	2027	FG296	ID	4	O (0-1)	xx	x	xx	tr	tr	—
124					I	—	xx	tr	—	—	—
125			C	1	W (0-5)	—	xx	tr	—	—	—
126		B48	IDP	1	O (0-2)	x	x	xx	—	—	—
127					I	—	xx	x	x	tr	—
II-c 128	2018	FG287-2	S	6	W (0-10)	xxx	—	xx	—	—	—
129		FG287-1	SP	6	W	xxx	—	xxx	—	—	—
130	2040	B54	S	6	W	xxx	—	xxx	tr	—	tr
131	2039	FG303	S	6	W (0-7)	xxx	—	xxx	—	—	—
132	2038	B53	S	6	W (0-7)	xxx	—	xxx	—	—	—
133	2037	FG302	S	6	W (0-12)	xxx	—	xxx	tr	—	—
134	2036	B52	IS	6	O (0-1)	xxx	—	xx	—	—	x
135					X (1-5)	x	x	x	—	—	—
136					X (5-15)	tr	x	x	tr	—	—
137					I (15-30)	—	xx	x	—	tr	—
138	2035	FG301	ID	4	O (0-3)	xx	tr	xx	—	—	—
139					I	tr	xx	x	tr	—	—
140	2017	FG286	ID	3	T (0-4)	x	xx	xx	tr	—	—
141					I	—	xx	xx	—	x	—

Table XIV-3 (continued)

Line	AN	St./Sample No.	Nodule sample			Mineral abundance					
			Shape	Surface	Part	TM	DM	Qtz	Ph	Pc	Mmt
142					B (0-1)	×××	×	×××	—	—	—
143		B42	1D	3	T (0-3)	×	××	×	—	—	—
144					I	—	××	×	tr	tr	—
145					B (0-1)	×××	×	××	—	—	tr
1)	Nodule shape	S: spherical, D: discoidal, I: irregular, P: polynucleate, C: crust.									
2)	Nodule surface	1: s, 2: s+s·r, 3: s+r, 4: s·r, 5: s·r+r, 6: r.									
3)	Analyzed part	W: whole, O: outermost, I: innermost, T: top, B: bottom. X: others. Depth from surface (in mm) is in parenthesis.									
4)	Minerals	TM: 10Å manganate, DM: 2 line form δ-MnO <sub>2</sub> (calculated), Qtz: quartz, Ph: phillipsite, Pc: plagioclase, Mmt: montmorillonite.									
5)	Abundance	×××: abundant, ××: common, ×: present, tr: traceable, —: not detected.									

composed of the δ-MnO<sub>2</sub> phase while rough surfaces (r) and intermediate surfaces (s·r) are always composed of 10 Å manganate. However, thickness of the layer of each mineral constituent on nodule surface is variable. Intermediate surface feature (s·r) consists of very thinly developed rough structure of the 10 Å manganate on smooth structure of the δ-MnO<sub>2</sub> phase. The chemical compositions of each powder sample are also remarkably consistent with mineral composition (USUI, Chapter XV in this cruise report).

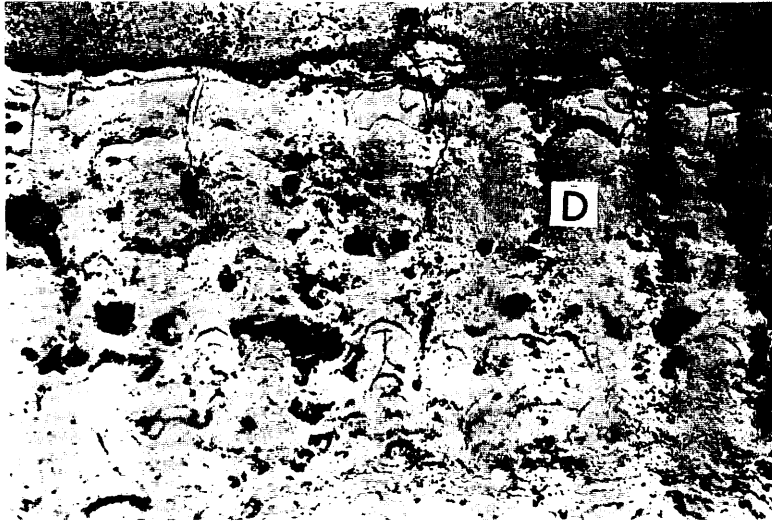
Silicate minerals are incorporated in nodules as fine particles and/or nuclei. Quartz is commonly incorporated in all samples and the range of variation is considerably small by up to a factor of two or three. The quartz contents appear to vary concordantly with nodule type and mineral composition. Nodule samples enriched in 10 Å manganate are also enriched in quartz, which may suggest dominant incorporation of detrital small particles of quartz from associated surface sediments into r-type nodules.

Phillipsite is most abundant inside s-type nodules. It is attributable to frequent occurrence of zeolitic claystone served as nodule nuclei.

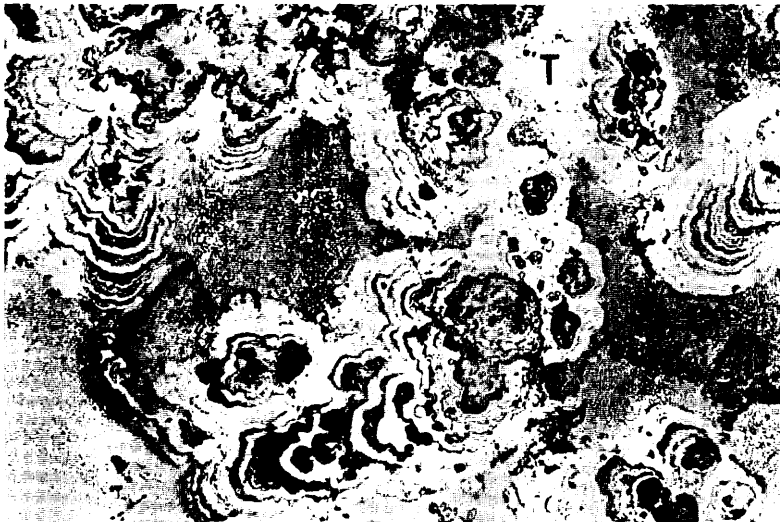
#### Variability of internal structure of nodules on detailed survey lines

Internal structure of nodules were described by means of megascopic and microscopic observations. Nodules consists principally of two ferromanganese constituents, with accessory manganese-free clayey silicate materials and nuclei.

Some workers proposed stratigraphic description of manganese nodules according to internal microstructure (SOREM and FOSTER, 1972; HEYE, 1975). I here proposed more definite criteria for nodule stratigraphy on the basis of mineralogy, which is more useful to description of internal structure of nodules and correlation between nodules. Two principal mineral phases, which exclusively consist representative microstructure, could be employed as stratigraphic units of nodules. Three units of ferromanganese materials are defined under reflecting microscope according to optical properties as follows:



(A)



(B)

Fig. XIV-1 Photomicrographs of typical stratigraphic units of manganese nodules. A)  $\delta$ - $MnO_2$  phase of stratified structure consisting smooth surface (D in photo), B) 10 Å manganate phase of cusped or dendritic structure consisting rough surface (T in photo).

- 1) 10 Å manganate phase: cusped or dendritic growth structure composing rough surface of nodules (Fig. XIV-1a),
- 2)  $\delta$ - $MnO_2$  phase: monotonously stratified or massive structure composing smooth surface of nodules (Fig. XIV-1b),
- 3) fine alternations of the above two phases sometimes showing intermediate

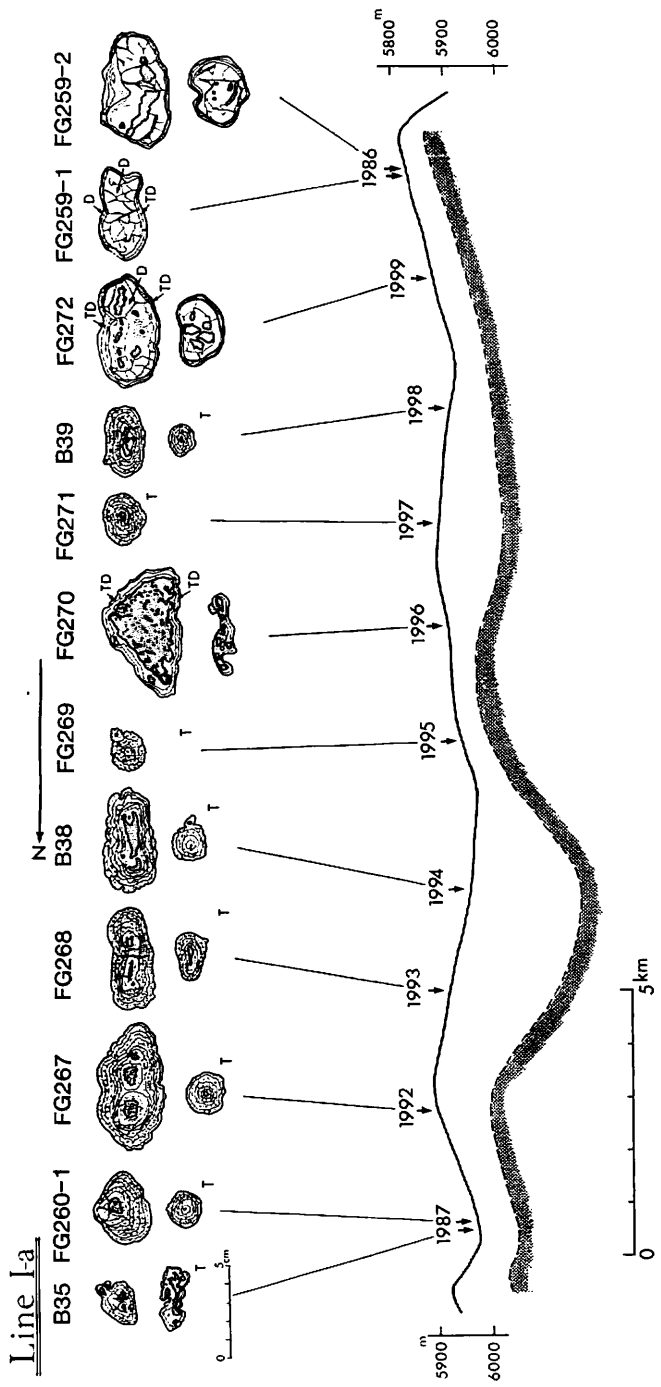


Fig. XIV-2 (1)

Fig. XIV-2 Variation of internal structure of manganese nodule on detailed survey lines. Shadows in topographic profiles denote opaque layers overlain by transparent layers in 3.5 kHz subbottom profiler. T, TD, DT, D denote manganese mineral abundance in this order.



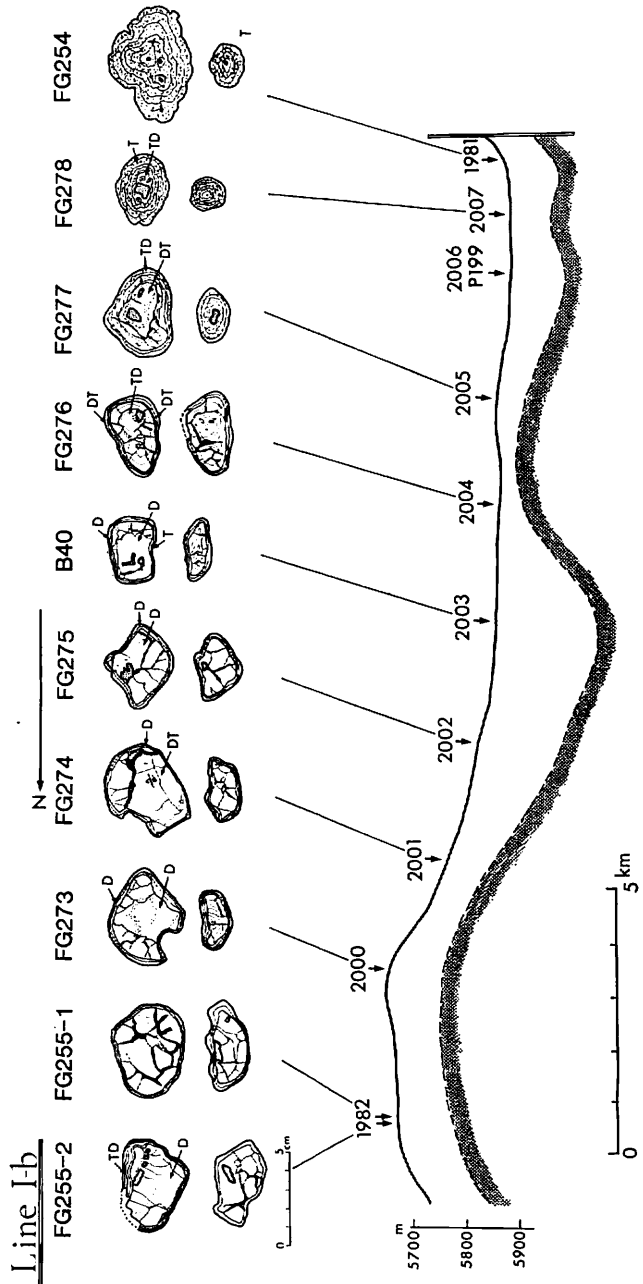


Fig. XIV-2 (2)

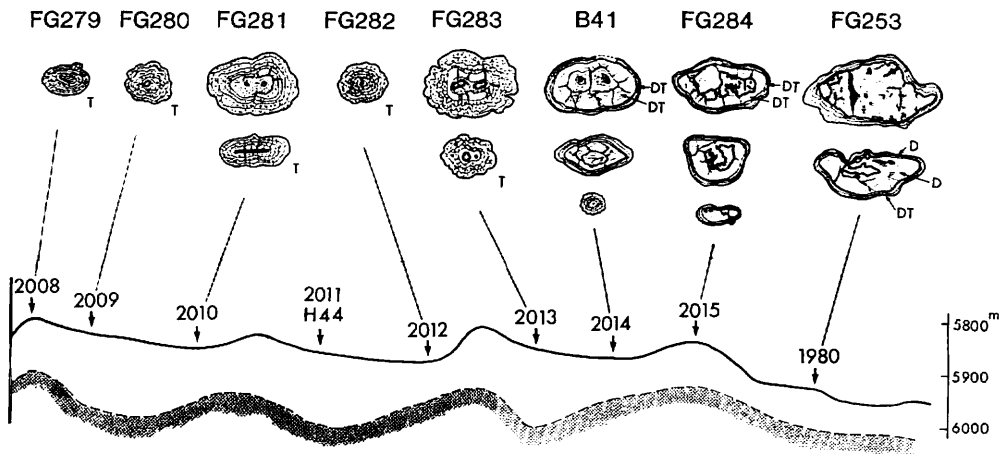


Fig. XIV-2 (3)

features as nodule surface, and another unit is nuclei composed of manganese-free material.

Megascopic description of polished nodule sections and related X-ray diffraction results are demonstrated in Figure XIV-2. Figure XIV-3 shows schematic stratigraphy of nodules along the surveyed lines.

The nodules frequently include conformable and unconformable boundaries between two phases. The unconformable boundary is characterized by sequential development of one unit of surrounding layer over the fragments of inside older nodules. This unconformable boundary is most dominant in smooth surface nodules of irregular shape. Internal older nodules often fragmented or fractured are composed of  $\delta$ - $MnO_2$ , while surrounding layers variable.

Along the detailed survey lines, nodule surface varies gradually or drastically from characteristic s-type to r-type nodules. However, the pattern of variation of internal structure is not always consistent with that of surface feature. On lines I-a and I-b the variation from types s to r are consistent with that of nodule internal structure. Type r is almost entirely composed of 10 Å manganate showing wholly concentric lamination, and type s is characterized by internal s-type nodules composed of  $\delta$ - $MnO_2$  phase and structure composing rough surface of nodules (Fig. XIV-1a). On the other hand, on lines II-a, II-b and II-c, above mentioned relationship is not the case. Type r sometimes contains older s-type nodules comprising large-size nodules near the transitional zones from smooth to rough nodules, e. g., Sts. 2033, 2036 etc.

Therefore the areas distribution of outer 10 Å manganate phase and that of internal s-type nodules are not always exclusive to each other but in some cases the former overlap the latter in the Area II nodules. As shown in Figure XIV-2, The variability of internal structure has no distinct relationship to the topography or subbottom structure on 3.5 kHz SBP records. Only possible relationships are the dominant development of the 10 Å manganate phase on nodules from deeper basin areas of flat topography and that of the  $\delta$ - $MnO_2$  phase inside nodules from topographic highs.

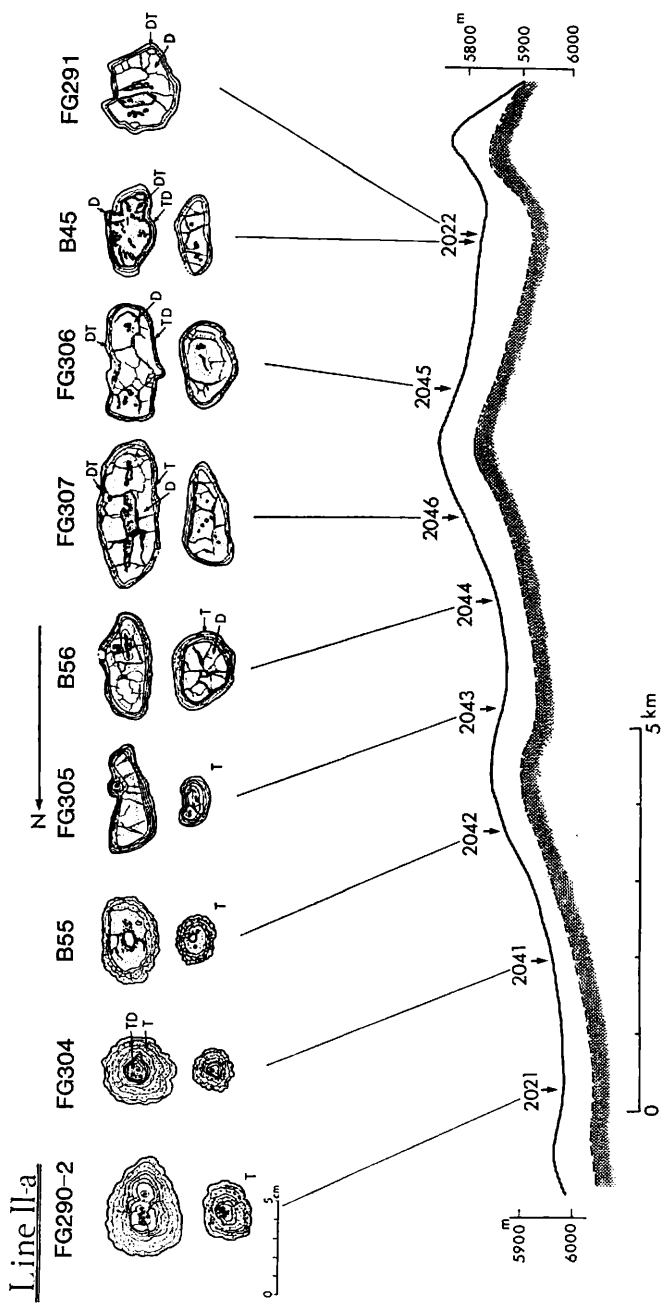


Fig. XIV-2 (4)

Line II-b

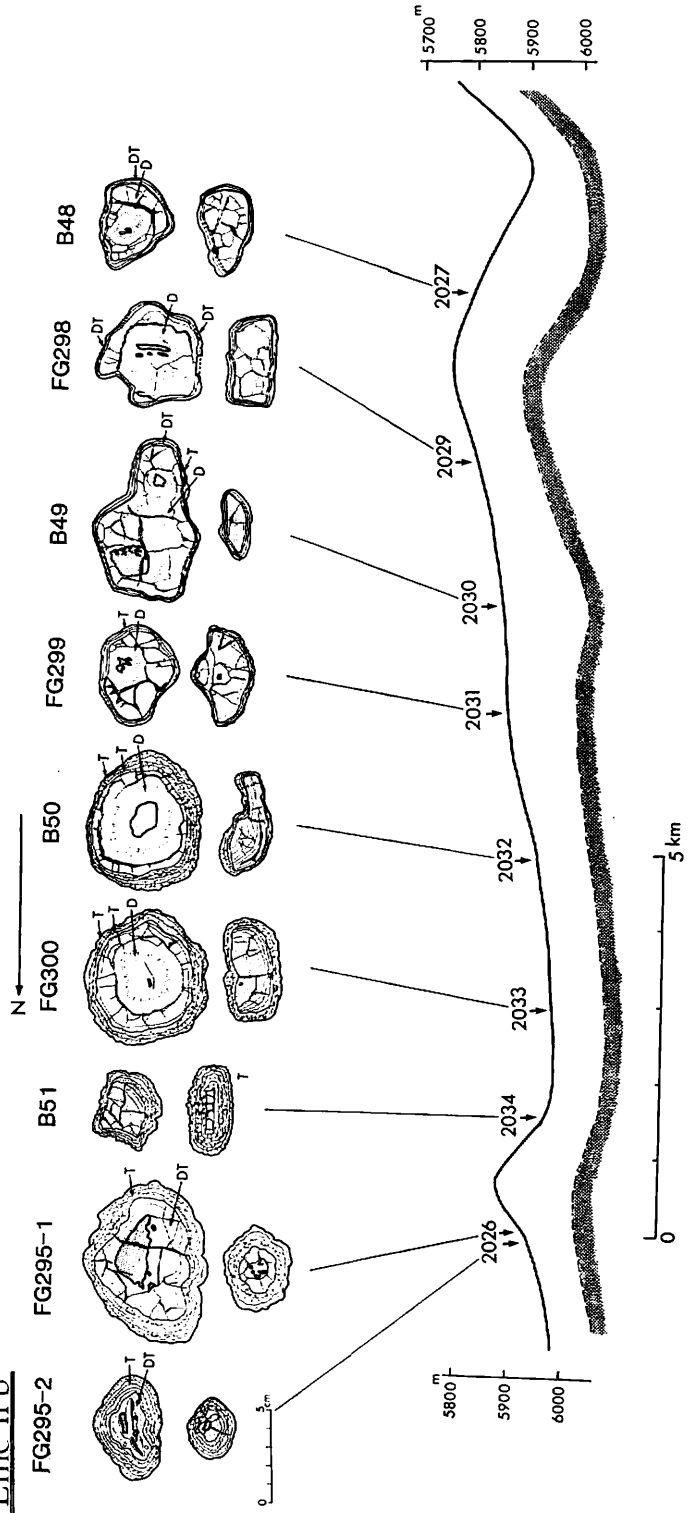


Fig. XIV-2 (5)

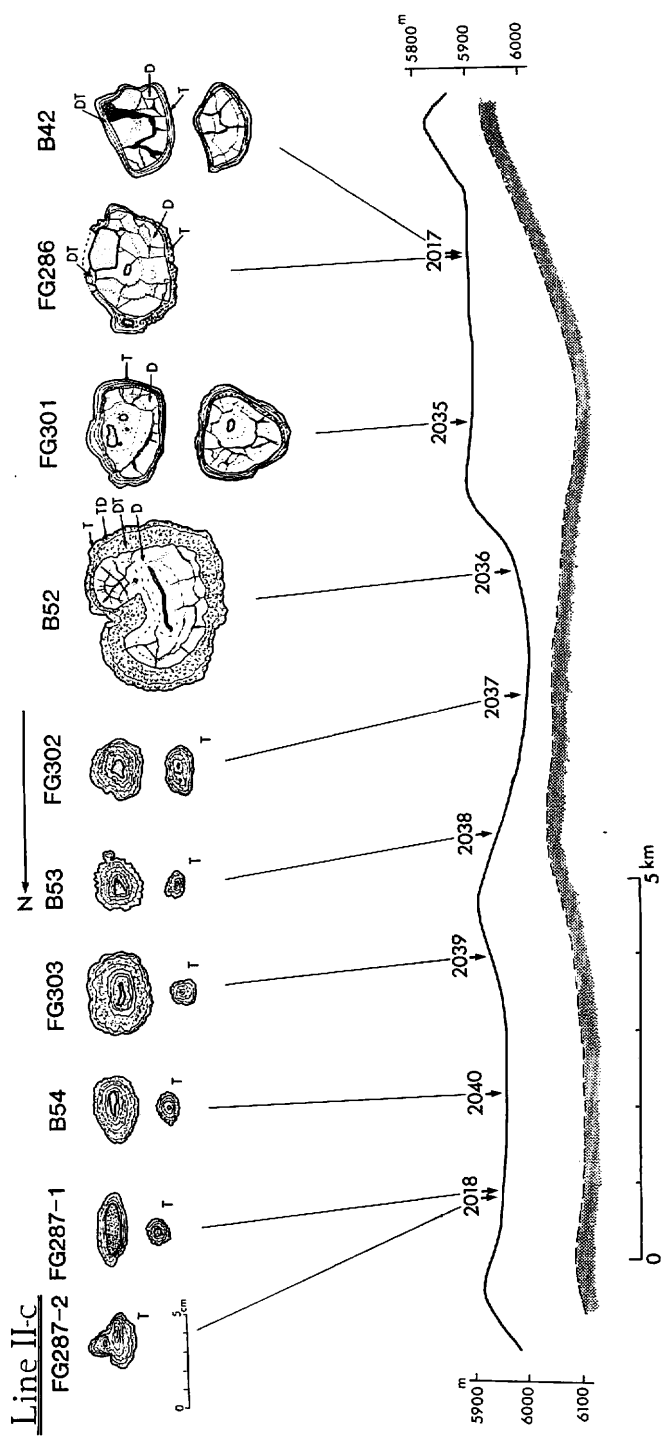


Fig. XIV-2 (6)

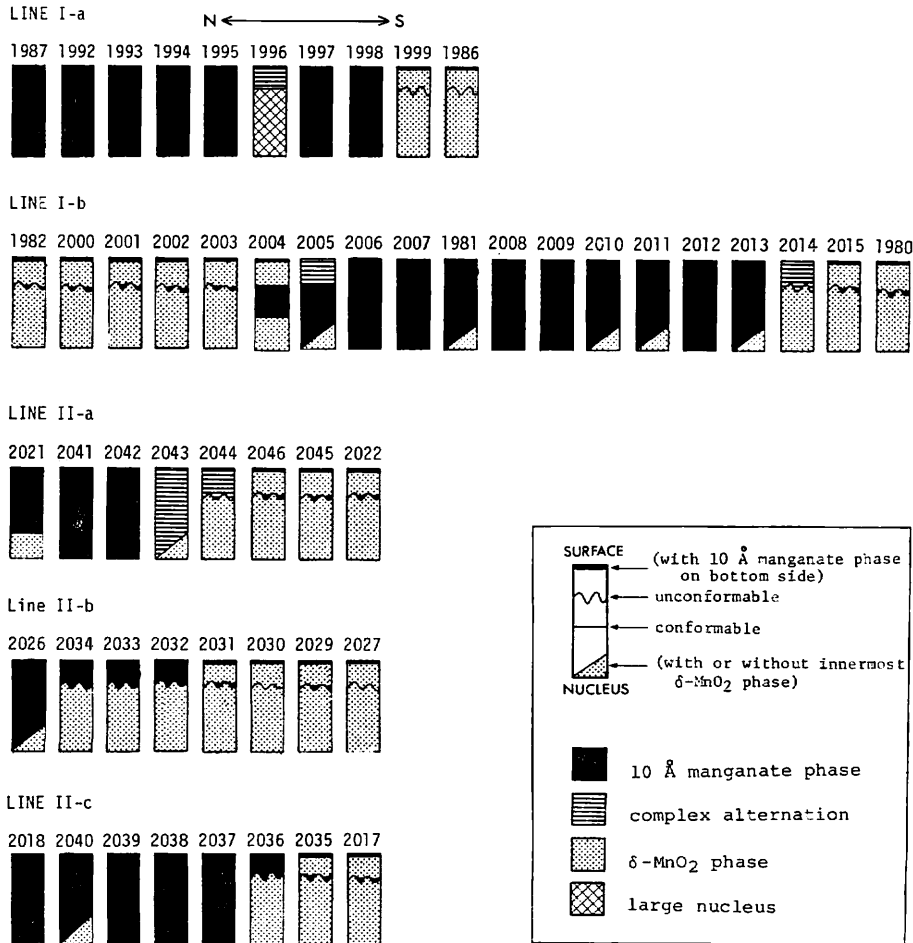


Fig. XIV-3 Schematic stratigraphy of manganese nodules interpreted by megascopic and microscopic observation of polished nodule sections and X-ray powder diffraction analyses.

Finely laminated unit (3) is dominantly observed on the nodule surface in the transitional zones from smooth to rough nodules, e. g., Sts. 1996, 2005, 2044 etc.

The older s-type nodule fragments in various nodules are significantly similar in microstructure and chemical composition (USUI and MOCHIZUKI, this cruise report) throughout the survey lines. The microstructure and chemical compositions of rough to intermediate surfaces composed of the 10 Å manganate are also fairly invariable despite morphological variability of nodules. This suggests a possibility of stratigraphic correlations of each phases between nodule samples. Schematic stratigraphy of manganese nodules reveals that deposition of the δ-MnO<sub>2</sub> is clearly divided into two facies; one is as internal older nodules occasionally broken and the other is outermost surrounding layers of s-type nodules. The former developed lower the unconformable boundary is dominantly fractured and fragmented while the latter developed over the

boundary is of less fractures. It is also revealed that the internal s-type nodules appear to be deposited in the older age than the surrounding layers of 10 Å manganate and typical type r entirely composed of 10 Å manganate.

To sum up, manganese nodules of this area are initiated with dominant and extensive deposition of the  $\delta$ -MnO<sub>2</sub> and zeolitic claystones, followed by the fragmentation of these nodules. After the fragmentation, deposition of  $\delta$ -MnO<sub>2</sub> and 10 Å manganate took place on the fragmented nodules or another nuclei. Considering that both phases are interbedded in nodules around the transitional zones from type s to r, the deposition of the two phases appear to be simultaneous. Difference of nodule surfaces related to mineral composition is probably due to local difference in sedimentary conditions.

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