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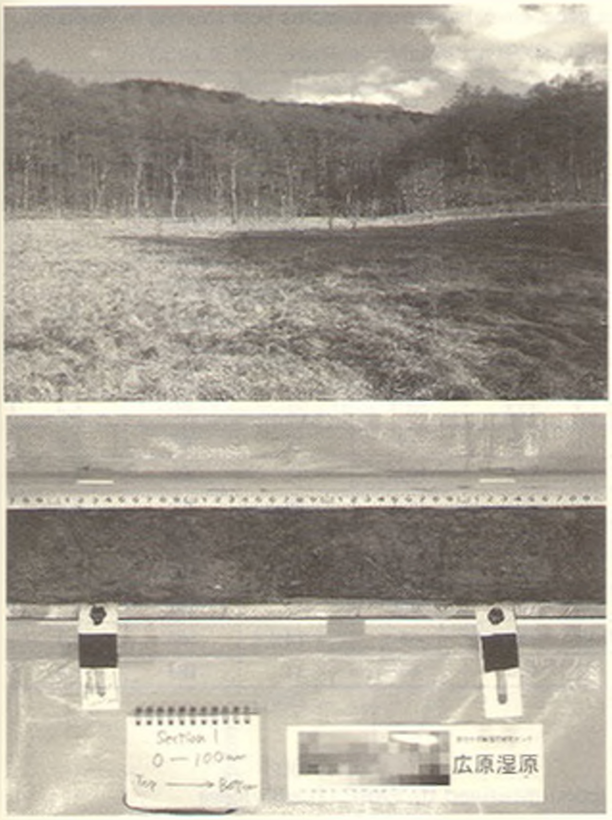
Natural Resource Environment and Humans

資源環境と人類

No. 5

2015. 3　明治大学黒耀石研究センター紀要



Photos View of Hiroppara peat bog (upper) and sediments core (lower)

Obsidian was globally used as an important material for stone tools from the prehistoric period. A numerous of obsidian sources range in the Japanese archipelago, and the central highland of Japan (Kirigamine Mountains) is a well-known obsidian source. Especially, the archeological studies in the area are advancing, and discuses obsidian sources, exploitations and transportations, in order to show the human migration and the cultural exchange since the Palaeolithic. Therefore, the central highland of Japan are suitable location to study for the long-term interaction between environment and human activities.

In the COLS, we take the research project in the central highland of Japan for better understanding the interaction between natural resource environment and humans.

In the project, I will conduct palaeoenvironmental investigations at the Hiroppara peat bog, in attempt to provide a useful information for the obsidian exploitation from the Palaeolithic to Jomon period in the area. This session will be based on our study site, and introduce the fundamental techniques in field and laboratory, to enhance your understanding for the palaeoenvironmental study.

Schedule and contents

1.1. Friday, 26, September ~ Field technique ~

In first day, main works are to take sediments cores in the peat bog, using the Hiller sampler (Slide-filling type). To understand present natural setting is the first step in the palaeoenvironmental studies. I will, therefore, expound on natural environment in the Kirigamine Mountains and Hiroppara peat bog. At
the bog, I will instruct a standard technique for drilling, such as selecting method of drilling point, the handling of drilling sampler. After that, the participants will try to taking a sediments cores using the sampler. The rough schedules at first day are as follows;

AM 8:00 Departure from COLS
AM 8:30 Arrive at Hiroppara
AM 9:00 Lecture for drilling
AM 9:30 Field work
------------AM 11:30~PM 0:30 Lunch --------------
PM 0:30 Field work
PM 2:30 Departure from Hiroppara

1.2. Friday, 27, September ~ Laboratory technique ~

In second day, we focus on laboratory techniques with particular pollen analysis. I will lecture how to sediments description and sub-sampling for radiocarbon dating and microfossil analyses. The participants will describe the lithological column for cores taken at the peat bog, and discuss about sedimentary process. Subsequently, I will introduce standard technique of pollen analysis, which is a typical method to reconstruct vegetation and climate changes. The participants prepare slides, and identify pollen fossils under the optical microscope. Also, we will discuss the vegetation and climate changes around the bog, based on your trial pollen identification. The rough schedules of second day are as follows;

AM 8:30 Lecture for sample observation at COLS
AM 9:00 Sample observation
------------AM 11:30~PM 0:30 Lunch --------------
PM 0:30 Lecture for pollen analysis
PM 1:00 Experiment and microscopic observation

2. Abstract of Presentation

2-1. Evgeny A. Nozdrachev

The use of portable XRF spectrometer Alpha-6000 (Innov-X Systems, Inc.) for studying obsidian artifacts from the Primorye Region and the Iturup Island, Russia

プリモーリェ地方とイトゥルップ島（ロシア）の黒曜石製石器研究に適用した携帯式XRF spectrometer Alpha-6000(Innov-X Systems, Inc.)の分析結果

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Table 1. PXRF measured element concentrations (ppm) for obsidian geochemical reference samples from sources in Hokkaido Island, Japan.

Sample Name (Number)	Obsidian Source	Analysis point	Zn	Pb	Rb	Sr	Y	Zr	Nb
JOR-1 (20101023)	Rubeshibe	1	44	20	114	118	25	127	4.3
		2	45	19	115	121	26	128	4.1
		3	47	21	119	125	26	128	5
		average	45	20	116	121	26	128	4.5
JOSH-1 (20111030-1)	Shirataki Hachigosawa	1	35	18	152	28.8	27	74	6.1
		2	29	18	151	27.8	26	76	5.3
		3	34	17	153	29.8	27	77	6.1
		average	33	18	152	28.8	27	76	5.8
JOSA-1 (20111031)	Shirataki Ajisai-notaki	1	30	17	172	10.7	31	66	6.4
		2	29	15	170	9.7	31	67	6.5
		3	32	19	176	10.6	32	69	6.2
		average	30	17	173	10.3	31	67	6.4
JOO-1 (20111101-1)	Oketo Kita-tokoroyama	1	22	20	139	65	24	105	5.1
		2	22	17	131	62	22	100	5.7
		3	22	19	136	64	23	102	5.3
		4	23	19	136	65	23	103	5.3
		average	22	19	136	64	23	103	5.4

Table 2. Comparison of element concentrations (ppm) measured by PXRF (1) with those obtained by ICP-MS (2) and WDXRF (3); after Yoshimitsu Suda et al.(2013) for ICP-MS and WDXRF data for the obsidian geochemical reference samples.

Sample	Zn			Pb			Rb			Sr		
Name (Number)	1	2	3	1	2	3	1	2	3	1	2	3
JOR-1 (20101023)	45	-	49	20	19	20	116	121	121	121	121	125
JOSH-1 (20111030-1)	33	-	35	18	16	18	152	159	159	29	30	30
JOSA-1 (20111031)	30	-	33	17	16	18	173	151	183	10	8	11
JOO-1 (20111101-1)	22	-	25	19	17	18	136	142	142	64	67	67

Sample	Y			Zr			Nb		
Name (Number)	1	2	3	1	2	3	1	2	3
JOR-1 (20101023)	26	26	27	128	126	123	5	6	5
JOSH-1 (20111030-1)	27	28	30	76	68	74	6	7	7
JOSA-1 (20111031)	31	31	35	67	50	66	6	6	7
JOO-1 (20111101-1)	23	24	25	103	98	99	5	6	6

Table 3. PXRF and ICP-MS measured element concentrations (ppm) for volcanic glasses from hyaloclastites from the Shkotovo basaltic plateau in Primorye (Russian Far East). ICP-MS analysis was conducted at the Analytical Center of the Far East Geological Institute, Far East Branch of the Russian Academy of Sciences, Vladivostok, Russia.

	Rb	Sr	Y	Zr	Nb
ICP-MS					
Sample 1	10	296	18	78	5
Sample 2	14	330	19	90	6
Sample 3	11	335	18	86	6
PXRF					
Sample 1	12	300	18	85	5
Sample 2	14	305	19	88	6
Sample 3	12	306	18	87	6

Table 4. The chemical composition (PXRF) of archaeological obsidians from the lower part of the Amur River basin. The samples are from the collection of I.Y. Shevkomud (the Museum of Archaeology, Khabarovsk Regional Museum after N.I. Grodekov, Khabarovsk, Russia).

Site	Zn	Pb	Rb	Sr	Y	Zr	Nb	Obsidian Source
Sample 3 Novotroitskoe 14	33	22	112	211	16	132	14	Samarga
Sample 2 Novotroitskoe 14	36	22	115	218	18	137	16	Samarga
Sample 4 Novotroitskoe 17	41	27	129	232	21	145	21	Samarga
Sample 1 Osinovaya Rechka 10	118	2	13	320	20	84	9	Shkotovo basaltic plateau

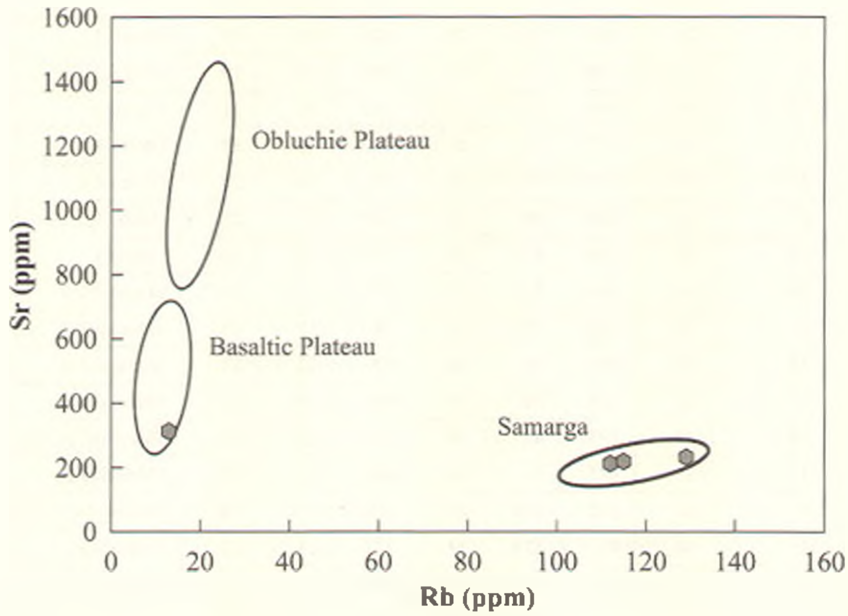

Fig.1 Bivariate plot of Rb vs. Sr concentrations for archaeological obsidians from the lower part of the Amur River basin. Source ellipces are derived from the work of Glascock et al. (2011) and corrected for data from Table 4.

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Implementation of combined geoarchaeological research it is possible only in case interdisciplinary studies that claim using different areas of knowledge. To date, all over the world the determining of the chemical composition of the archaeological artefacts is conducted by using portable x-ray fluorescence spectrometers. The main advantages of these spectrometers are nondestructive way of doing analysis, small analysis area (typically not more than 10 mm) and their small size for field work.

In present work some example using portable x-ray fluorescence spectrometer Alpha-6000 (Innov-X Systems, Inc.) for the analysis of obsidian artefacts are presented. The spectrometer consists of stable tantalum anode x-ray tube, thermoelectrically cooled Si-PIN X-ray detector with energy resolution 200 eV at 5.9 keV for 19 μs peaking time. Spectrometer is driven by the HP iPAQ pocket PC running under Microsoft Windows Mobile 5.

Analysis is done at 40 kV and 15 μA, with an aluminium primary beam filter 2 mm thick. The analysed obsidian artefacts must have more than 6-8 mm in smallest dimension and 2 mm thick. Each sample at specified point is shot four times for 180 seconds each, which produced a combined analytical readings. These readings are corrected for characteristic line overlaps and converted to concentrations of Zn, Pb, Rb, Sr, Y, Zr, Nb by means of external software Microsoft Office Excel.

Unfortunately the proprietary software Innov-X only includes simple empirical model that doesn't give possibility to correct matrix effects and line overlaps. For our work we used Soil mode of this software that includes matrix correction

Table 5. The chemical composition (PXRF) of archaeological obsidians from the Yankito 2 site on Iturup Island. The obsidian artifacts were collected during archaeological excavations of ancient settlement on the Yankito 2 site on Iturup Island (Kuril District of the Sakhalin Region, Russia) by the joint expedition of the Sakhalin Regional Museum and the Peter the Great Museum of Anthropology and Ethnography (Kunstkamera) under the scientific direction of senior researcher O.V. Yanshina in july 2013 year.

Sample Number	Zn	Rb	Sr	Y	Zr	Nb	Pb	Obsidian Source
2	26	145	70	25	108	11	22	Oketo-1
3	28	156	75	28	113	15	21	Oketo-1
4	24	142	67	24	102	9	19	Oketo-1
6	26	148	71	27	110	11	21	Oketo-1
7	31	166	79	30	117	19	22	Oketo-1
8	28	155	76	29	116	16	21	Oketo-1
9	27	148	71	26	108	13	19	Oketo-1
15	27	156	76	24	115	16	21	Oketo-1
16	26	147	71	25	107	12	19	Oketo-1
20	27	157	76	29	113	16	20	Oketo-1
21	32	166	80	31	119	19	22	Oketo-1
22	29	166	81	32	120	20	24	Oketo-1
10	34	163	32	31	79	11	21	Shirataki-A
11	38	162	32	30	78	11	19	Shirataki-A
12	45	191	40	38	93	22	27	Shirataki-A
17	42	184	38	36	89	18	22	Shirataki-A
18	45	195	41	39	94	22	25	Shirataki-A
13	33	183	11	34	66	11	18	Shirataki-B
1	42	122	121	26	113	7	24	Rubeshibe
5	50	87	220	29	177	24	22	Unknown
14	61	230	51	48	108	34	34	Unknown
19	47	212	103	44	145	35	35	Unknown
23	65	234	53	51	110	35	38	Unknown

by normalizing the count rates of characteristic radiation to background scattering in a range of energy from 19.8 to 20.3 keV. In the Soil mode we have added elements Y, Nb and corrected a range of energy for all chemical elements under study: Zn 8.42-8.75 keV, Pb 12.42-12.70 keV, Rb 13.16-13.54 keV, Sr 13.89-14.36 keV, Y 14.71-15.11 keV, Zr 15.49-15.96 keV, Nb 16.40-16.80 keV. We also have changed internal constants so-called "Element Rates" of the software: the sensitivity factor and offset. These constants and line overlap coefficients have been individually adjusted for each element by means of iterative analysis of pressed powder pellets with known concentrations of the elements. The pellets (40 mm in diameter) were prepared by pressing 7 gramm of selected rock powders or reference rock standards mixed with a few drops of 6 % aqueous solution of polyvinyl alcohol. As the claimed element concentrations we used recommended values for reference samples or values determined on a wavelength dispersive X-ray fluorescence spectrometer S4 Pioneer (Bruker AXS, Germany) on the same above mentioned pellets. Such approach with hard binding of acquired concentrations to element concentrations in pressed pellets and carefully fitted line overlaps coefficients give possibility to obtain absolute element concentrations in obsidian artifacts.

It's the first time to study chemical composition of volcanic glasses by means of portable x-ray fluorescence spectrometer Alpha-6000 (PXRF). Test analysis of four obsidian geochemical reference samples from sources in Hokkaido Island was conducted in order to validate the accuracy of this technique. The samples are from the collection of V.K. Popov (the Far East Geological Institute, Far East Branch of the Russian Academy of Sciences, Vladivostok, Russia). These obsidian reference samples

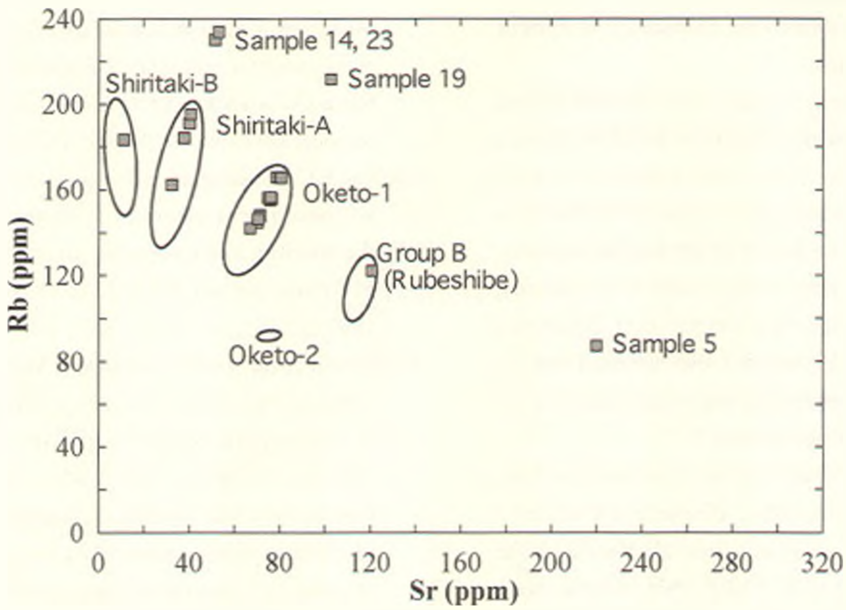

Fig.2 Bivariate plot of Sr vs. Rb for archaeological obsidians from the Yankito 2 site on Iturup Island. Source ellipces are derived from the work of Phillips and Speakman (2009) and corrected for data from Table 5.

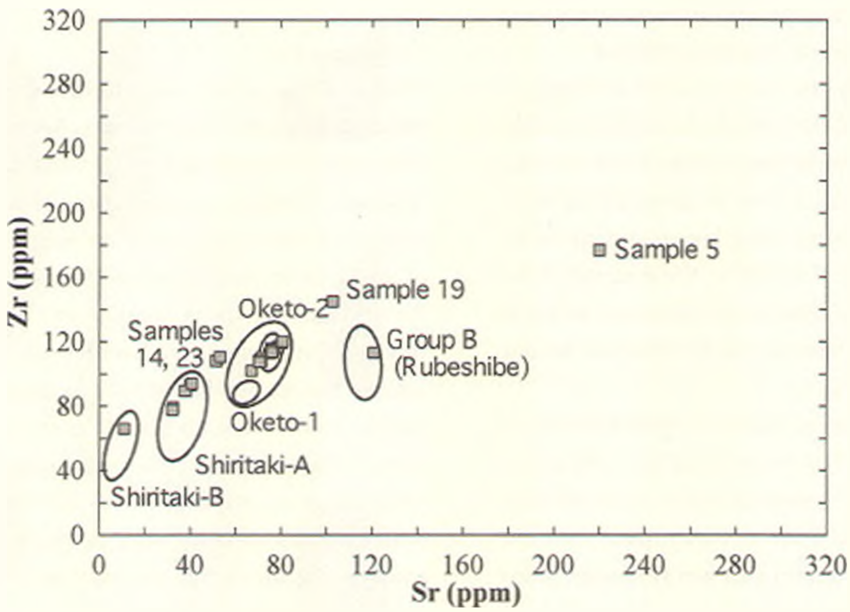

Fig.3 Bivariate plot of Sr vs. Zr for archaeological obsidians from the Yankito 2 site on Iturup Island. Source ellipces are derived from the work of Phillips and Speakman (2009) and corrected for data from Table 5.

are named JOR-1: Japanese Obsidian Rubeshibe. JOSH-1: Japanese Obsidian Shirataki Hachigosawa. JOSA-1: Japanese Obsidian Shirataki Ajisai-notaki, JOO-1: Japanese Obsidian Oketo. The results of such analysis are given in Table 1. The represented average element concentrations were compared with those obtained by ICP-MS at the Institute of the Earth's crust, Siberian Branch of the Russian Academy of Sciences, Irkutsk, Russia and by WDXRF at Center for Obsidian and Lithic Studies, Meiji University, Nagano Prefecture, Japan (Yoshimitsu Suda et al., 2013). The result of this compilation is given in Table 2. The comparison of PXRF analysis with ICP-MS and WDXRF has shown their consistency in terms of obsidian source identification.

Similarly, for comparative study, three volcanic glasses from hyaloclastites from the Shkotovo basaltic plateau in Primorye (Russian Far East) were analysed by both PXRF and ICP-MS at the Analytical Center of the Far East Geological Institute, Far East Branch of the Russian Academy of Sciences, Vladivostok, Russia. The results of the analysis, given in Table 3, have shown no significant difference between PXRF and ICP-MS data and also indicated that the PXRF is accurate and reproducible analytical technique for quantitative analysis of volcanic glasses.

In the sequel, the archaeological obsidians from the lower part of the Amur River basin (Osinovaya Rechka 10 and Novotroitskoe 14,17 sites) and from the Yankito 2 site on Iturup Island were studied by PXRF. These samples were provided by the courtesy of I.Y. Shevkomud (the Museum of Archaeology, Khabarovsk Regional Museum after N.I. Grodekov, Khabarovsk, Russia), O.A. Shubina (the Sakhalin Regional Museum, Yuzhno-Sakhalinsk, Russia) and O.V. Yanshina (the Peter the Great Museum of Anthropology and Ethnography (Kunstkamera), St. Petersburg, Russia).

In order to identify the sources of archaeological obsidians from the sites of the Amur River basin the mutual correlation between chemical composition of the samples under study and volcanic glasses from Primorye and the Amur River basin was conducted by using binary diagram Sr-Rb from the work of Glascock et al. (2011). It was identified that the samples have chemical composition equivalent to that of volcanic glasses from the Samarga and the Shkotovo basaltic plateau sources (Table 4; Fig. 1).

The bivariate plots Rb-Sr and Zr-Sr from the work of Phillips and Speakman (2009) were used for comparative analysis of trace element concentrations in archaeological obsidians from the Yankito 2 site on Iturup Island and those in volcanic glasses from Kamchatka and Hokkaido Island sources. In the result of the research four geologic sources located in Hokkaido Island were identified for the obsidian artifacts (Table 5; Fig. 2, 3). We have established that "unknown" obsidian samples of the group B in the work of Phillips and Speakman (2009) would correspond to the Rubeshibe source (Fig. 2, 3).

On the whole these conclusions about original sources of archaeological obsidians from the Amur River basin and the Kuril Islands are illustrated in fig. 1-3.

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2-2. Wugan Luo

Provenance study on obsidian artifacts from Helongdadong Site, Jilin Province, Northeast China

中国吉林地方の大洞遺跡出土の黒曜石製石器の産地研究

Abstract: Portable energy-dispersive X-ray fluorescence (PXRF) has been used to test the main and trace elements of obsidian artifacts from the Helongdadong Site. And the obsidian ores from the northern Korea, Nagano and Hokkaido in Japan have also been analysed. The results indicate that the obsidian artifacts in different types, such as blades, microblades, microblade cores, scrappers, carvers, points, bifaces and the processing pieces share the similar characteristics. It indicates that the obsidian artifacts in different types share the same provenance, and they were made in the local. And we noticed the special type of carvers from the sites in the north and middle Japan, the Huangwu type bifaces. The obsidian carvers of Huangwu type bifaces from the Helongdadong Site share the same trace elements characteristics with the other artifacts excavated in Helongdadong, but it is different from the obsidian carvers of Huangwu type bifaces from Japan. It indicates that the obsidian carvers of Huangwu type bifaces from the Helongdadong Site and Japan used different source of obsidian ores. Further study shows that the obsidian artifacts from Helongdadong Site share the same similar trace elemental characteristics with the obsidian ores from the northern Korea. Through investigation, we know the obsidian