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U–Pb age of the *Pachydiscus flexuosus* Zone (Maastrichtian, Cretaceous) in the Nakatonbetsu area, Hokkaido, northern Japan

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Abstract Radiometric age dating of zircons ($^{238}U/^{206}Pb$ ratios, using the LA-ICP-MS method) contained in a tuff sample obtained from the middle part of the *Pachydiscus flexuosus* Zone (taxon-range zone) of the Yezo Group in the Nakatonbetsu area, Hokkaido, northern Japan, yielded an age of 69.8 ± 0.8 Ma, 95% conf., which suggests a late early to early middle Maastrichtian (latest Cretaceous) age under the three-subdivision scheme for the Maastrichtian. This result combined with previous magnetostratigraphic correlation indicates that the middle and upper parts of the *P. flexuosus* Zone span the entire middle Maastrichtian, while the lower part containing *Gaudryceras hamanakense* or *G. tombetsense* is probably a correlative of the upper lower Maastrichtian.

Key words: Cretaceous, Maastrichtian, *Pachydiscus flexuosus* Zone, U–Pb age, zircon

Introduction

Pachydiscus is one of the most common ammonoid genera in Maastrichtian strata in the Northwest Pacific region and several species have been described from Japan and the Russian Far East (Matsumoto, *et al.*, 1979; Matsumoto and Morozumi, 1980; Yazikova, 1994; Maeda *et al.*, 2005). Because of its successive stratigraphic occurrences and the restricted stratigraphic ranges of its many species, *Pachydiscus* is considered to be an ideal ammonoid genus for precise biostratigraphic correlation of Maastrichtian strata in this particular region (Toshimitsu *et al.*, 1995; Shigeta *et al.*, 2015).

Based on the two-subdivision scheme for the Maastrichtian (Toshimitsu *et al.*, 1995), the *Pachydiscus flexuosus* Zone was considered to be an approximate correlative of the upper Maastrichtian, but Shigeta *et al.* (2015) demonstrated that *P. flexuosus* Matsumoto in Matsumoto *et al.*, 1979 occurs in the upper portion of the lower part as well as the middle part of the Senposhi Formation in eastern Hokkaido. These sediments correlate with polarity chron C31n, i.e., middle to upper part of the middle Maastrichtian under the three-subdivision scheme of approximate equal duration, which we also follow in this paper. Unfortunately, the lowermost part of the Senposhi Formation and the underlying formation are not fossiliferous, so it is not known to which polarity chron the lower limit of the stratigraphic distribution of *P. flexuosus* should be assigned.

The Northwest Pacific region contains three continuous, well-studied Maastrichtian successions containing *Pachydiscus flexuosus* as follows: the Krasnoyaruka Formation in the Makarov area, southern Sakhalin, Russian Far East (Maeda *et al.*, 2005), the Heitaro-zawa Formation in the Nakatonbetsu area, northern Hokkaido, northern Japan (Ando *et al.*, 2001), and the Senposhi Formation in eastern Hokkaido (Nifuku *et al.*, 2009; Shigeta *et al.*, 2015). Magnetostratigraphy in the first two formations has never been studied, but both are known to contain thick tuff beds.

In this paper, we utilize zircon-based geochronology to calibrate the international correlation of the *Pachydiscus flexuosus* Zone in the Heitaro-zawa Formation in the Nakatonbetsu area, northern Hokkaido, and establish a more precise global correlation of the zone.

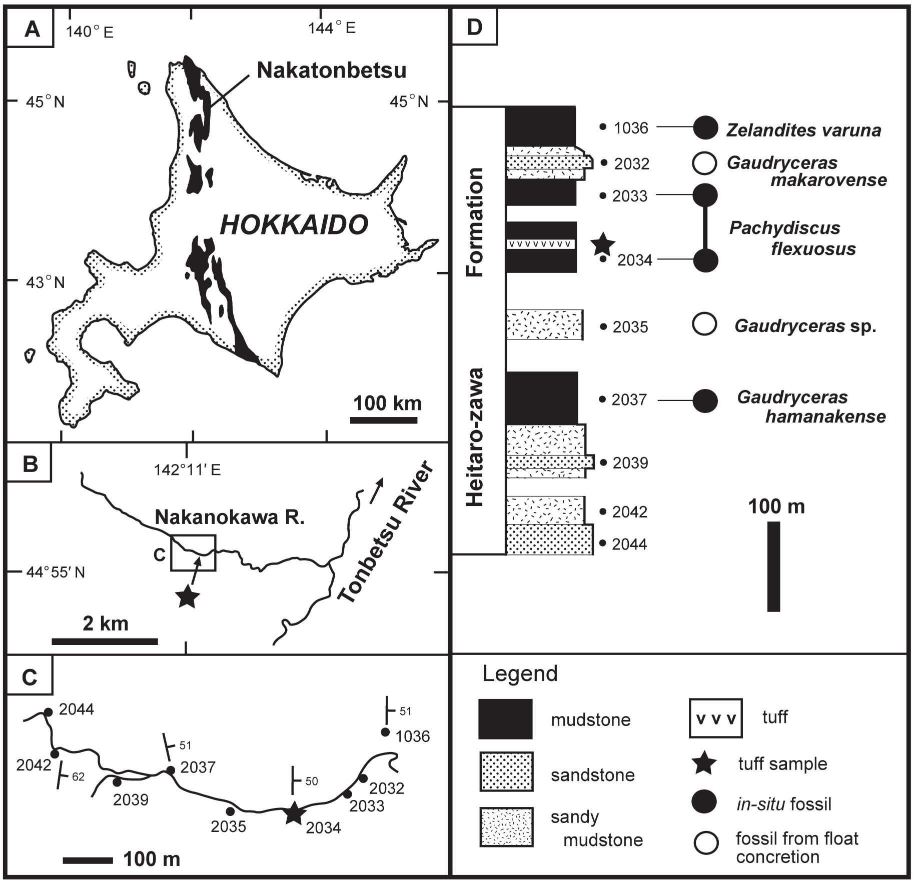

Fig. 1. Index map showing distribution of the Yezo Group (black areas) in Hokkaido (A), locality (B, C) and stratigraphic section (D) showing the position of the tuff bed that provided the U–Pb zircon age, and the occurrence of important ammonoids in the Heitaro-zawa Formation along the Nakanokawa River in the Nakatonbetsu area, Hokkaido, northern Japan.

Notes on stratigraphy

A continuous succession of the Heitaro-zawa Formation, the uppermost part of the Yezo Group, is well exposed along several western tributaries of the Tonbetsu River in the Nakatonbetsu area, northern Hokkaido (Igi, 1959; Matsumoto *et al.*, 1980, 1981; Ando *et al.*, 2001). Because the formation yields numerous well-preserved Maastrichtian megafossils from various horizons, this area is regarded as having extremely high potential for the study of Maastrichtian biostratigraphy in the Northwest Pacific region (Toshimitsu *et al.*, 1995; Shigeta and Nishimura, 2013; Shigeta *et al.*, 2015).

The 500–1000 m thick Heitaro-zawa Formation consists mainly of sandy mudstone and mudstone, intercalated with 1–50 cm thick white, vitric tuff beds (Ando *et al.*, 2001). Ammonoids typical of the early Maastrichtian, i.e., *Gaudryceras hobetsense* Shigeta and Nishimura, 2013 and *Damesites hetonaiensis* Matsumoto, 1954 (Shigeta and Nishimura, 2013), occur in the lower part of the formation, while *G. tombetsense* Matsumoto, 1984 and *Pachydiscus flexuosus* (Matsumoto, *et al.*, 1979) are found in the lower middle part and upper middle part, respectively. The occurrence of *Zelandites varuna* (Forbes, 1846) in the upper part correlates the strata with the lower part of the upper Maastrichtian (Shigeta *et al.*, 2015). The formation is unconformably overlain by the upper Paleocene Oku-utsunai Formation (Ando *et al.*, 2001). Uppermost Maastrichtian to lower Paleocene strata, including the Cretaceous/Paleogene boundary, has presumably been removed by erosion (Shigeta *et al.*, 2015).

Material and methods

Material: Radiometric age analysis was conducted on zircon grains extracted from a tuff sample taken from a 50 cm thick white, vitric tuff bed in the *Pachydiscus flexuosus* Zone at loc. 2034 along the Nakanokawa River, a western tributary of the Tonbetsu River (44°55′12.95″N, 142°11′07.90″E; Fig.

Table 1. LA-ICP-MS analyzed data and calculated ages of zircon contained in a tuff sample from the Heitaro-zawa Formation. Errors are 1 sigma. Pb_c and Pb^* indicate the common and radiogenic portions, respectively. "*" with label means the data are discordant.

Labels	$^{206}Pb_c$ $^{(1)}$ (%)	U (ppm)	Th (ppm)	Th/U	$^{238}U/^{206}Pb^*$ $^{(1)}$	$^{207}Pb^*/^{206}Pb^*$ $^{(1)}$	$^{238}U/^{206}Pb^*$ age $^{(1)}$ (Ma)	$^{238}U/^{206}Pb^*$ age $^{(2)}$ (Ma)
NK_01.1	0.58	272	263	0.99	95.79 ± 2.44	0.0454 ± 0.0101	66.9 ± 1.7	67.1 ± 1.6
NK_02.1	0.00	246	154	0.64	90.92 ± 2.05	0.0417 ± 0.0036	70.5 ± 1.6	70.5 ± 1.6
NK_03.1	0.00	165	120	0.75	25.03 ± 0.42	0.0521 ± 0.0025	252.6 ± 4.2	252.3 ± 4.3
NK_04.1	2.41	193	137	0.73	87.51 ± 2.42	0.0437 ± 0.0108	73.2 ± 2.0	73.6 ± 1.9
NK_05.1	0.00	136	92	0.70	86.94 ± 2.34	0.0411 ± 0.0048	73.7 ± 2.0	73.7 ± 2.0
NK_06.1	0.00	92	60	0.67	91.09 ± 3.53	0.0545 ± 0.0073	70.4 ± 2.7	69.8 ± 2.8
NK_07.1	1.15	124	83	0.69	95.23 ± 3.02	0.0539 ± 0.0123	67.3 ± 2.1	66.8 ± 2.1
NK_08.1	0.00	206	117	0.58	90.49 ± 2.38	0.0417 ± 0.0044	70.8 ± 1.9	70.8 ± 1.9
NK_09.1	0.00	237	211	0.91	89.41 ± 2.52	0.0545 ± 0.0046	71.7 ± 2.0	71.1 ± 2.0
NK_10.1	1.79	233	211	0.93	90.57 ± 2.63	0.0572 ± 0.0117	70.8 ± 2.0	69.9 ± 1.9
NK_11.1	2.71	129	75	0.60	97.65 ± 3.83	0.0265 ± 0.0126	65.7 ± 2.6	67.4 ± 2.5
NK_12.1	0.00	203	142	0.72	92.20 ± 2.27	0.0438 ± 0.0047	69.5 ± 1.7	69.5 ± 1.7
NK_13.1	0.00	531	975	1.88	94.12 ± 1.64	0.0513 ± 0.0025	68.1 ± 1.2	67.8 ± 1.2
NK_14.1	2.53	201	182	0.93	91.49 ± 2.49	0.0400 ± 0.0114	70.1 ± 1.9	70.7 ± 1.8
NK_14.2	0.00	174	118	0.70	90.37 ± 2.40	0.0480 ± 0.0049	70.9 ± 1.9	70.9 ± 1.9
NK_15.1	0.39	180	99	0.57	92.35 ± 2.57	0.0374 ± 0.0083	69.4 ± 1.9	69.7 ± 1.8
NK_16.1	0.37	424	207	0.50	33.29 ± 0.50	0.0463 ± 0.0031	190.8 ± 2.8	191.5 ± 2.8
NK_17.1	0.38	369	321	0.89	90.97 ± 1.91	0.0479 ± 0.0067	70.5 ± 1.5	70.4 ± 1.4
NK_18.1	0.09	249	191	0.78	92.95 ± 2.70	0.0465 ± 0.0082	69.0 ± 2.0	69.0 ± 1.9
NK_19.1	3.99	274	221	0.83	93.50 ± 2.51	0.0355 ± 0.0128	68.6 ± 1.8	69.6 ± 1.7
NK_20.1	0.83	150	98	0.67	92.58 ± 3.10	0.0411 ± 0.0099	69.3 ± 2.3	69.8 ± 2.3
NK_21.1	0.00	297	266	0.92	86.78 ± 1.80	0.0535 ± 0.0039	73.9 ± 1.5	73.3 ± 1.6
NK_22.1	0.00	233	197	0.87	93.71 ± 2.49	0.0546 ± 0.0051	68.4 ± 1.8	67.8 ± 1.8
NK_23.1	0.00	423	590	1.43	92.97 ± 1.62	0.0439 ± 0.0034	69.0 ± 1.2	69.0 ± 1.2
NK_24.1	0.00	458	993	2.22	94.58 ± 1.90	0.0501 ± 0.0029	67.8 ± 1.4	67.6 ± 1.4
NK_25.1*	0.00	419	689	1.69	92.96 ± 1.71	0.0584 ± 0.0037	69.0 ± 1.3	68.0 ± 1.3
NK_26.1	1.07	251	183	0.75	89.62 ± 2.16	0.0441 ± 0.0074	71.5 ± 1.7	71.8 ± 1.7
NK_27.1	3.31	234	198	0.87	91.76 ± 2.69	0.0385 ± 0.0120	69.9 ± 2.0	70.7 ± 1.9

(1) Common Pb corrected by assuming $^{206}Pb/^{238}U$–$^{208}Pb/^{232}Th$ age-concordance
(2) Common Pb corrected by assuming $^{206}Pb/^{238}U$–$^{207}Pb/^{235}U$ age-concordance

1).

Methods: Zircon grains were extracted by standard techniques: crushing, heavy liquid separation and handpicking. Then, the zircon grains were mounted in an epoxy disc with the FC1 zircon standard ($^{206}Pb/^{238}U = 0.1859$; Paces and Miller, 1993), and NIST SRM610 glass standard and polished until the center of each grain was exposed. The backscattered electron and cathodoluminescence images of the zircon grains were used for selection of the sites for analysis. U–Pb dating of the sample was carried out using the LA-ICP-MS method, which consists of a NWR213, a 213 nm wave length Nd-YAG laser ablation system (Elemental Scientific Lasers) and an Agilent 7700x quadrupole ICP-MS (Agilent Technologies), installed at the National Museum of Nature and Science at Tsukuba, Japan. The experimental conditions and procedures for the measurements were based on the methods described in Tsutsumi *et al.* (2012). The spot size of the laser was 25 μm. Correction for common Pb was made on the basis of the measured $^{207}Pb/^{206}Pb$ ratio (^{207}Pb correction) or $^{208}Pb/^{206}Pb$ and Th/U ratios (^{208}Pb correction; e.g. Williams, 1998) as well as the model for common Pb compositions proposed by Stacey and Kramers (1975). In this paper, we adopt the ^{207}Pb correction for age discussion because it is more effective in calculating the Phanerozoic ^{238}U–$^{206}Pb^*$ age than the ^{208}Pb correction (e.g. Williams, 1998). The pooled ages presented in this study were calculated using Isoplot/Ex software (Ludwig, 2003). The uncertainties in the mean ^{238}U–$^{206}Pb^*$ ages represent 95% confidence intervals (95% conf.). $^{206}Pb^*$ indicates radiometric ^{206}Pb.

Results

Analyzed data from the zircons, including common ^{206}Pb content rate in total Pb, U, and Th concentrations, Th/U, $^{238}U/^{206}Pb^*$, and $^{207}Pb^*/^{206}Pb^*$ ratios, and radiometric $^{238}U/^{206}Pb^*$ ages are listed in Table 1. All errors state 1 sigma level. All zircons in

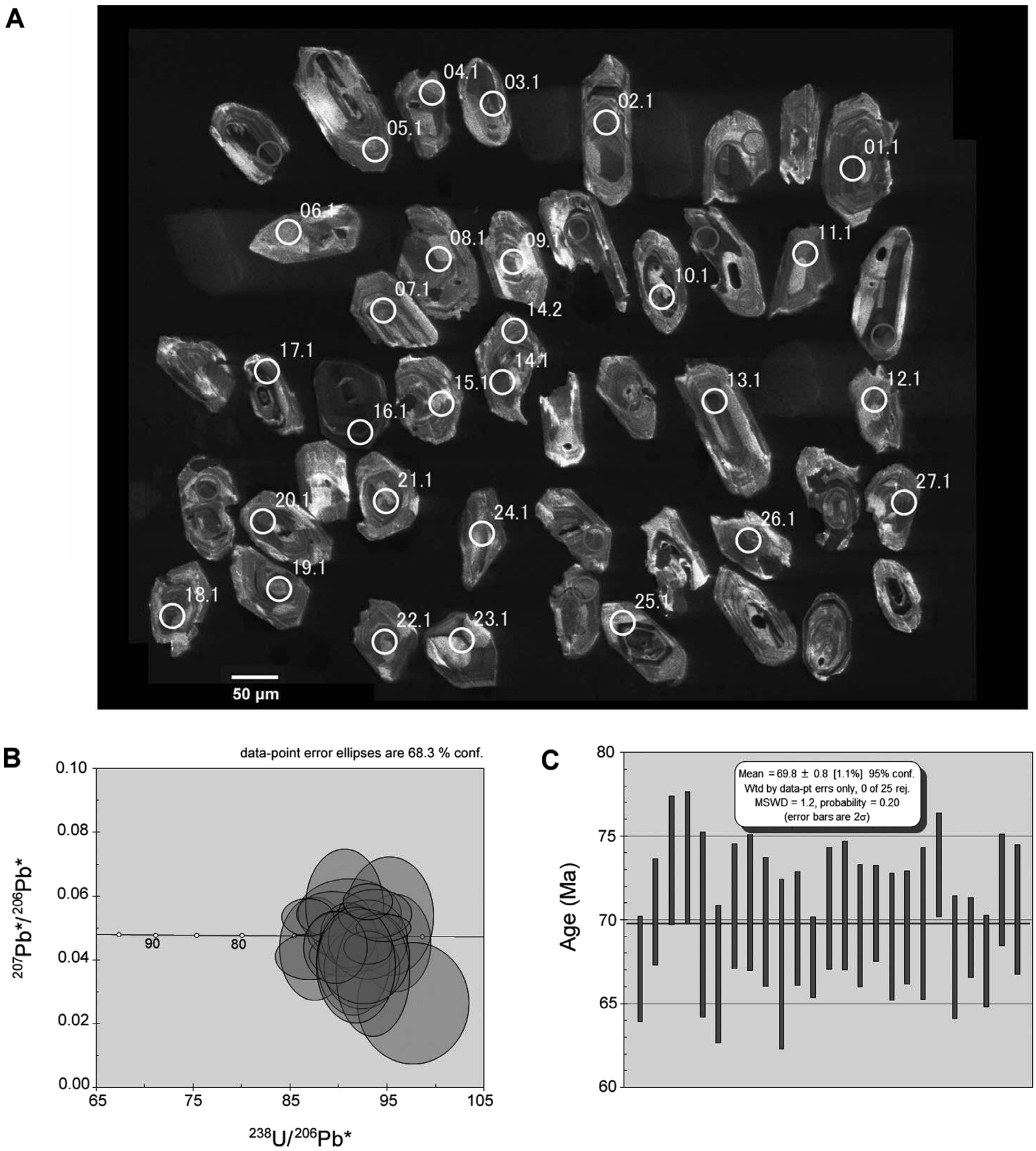

Fig. 2. U–Pb ages of zircon grains contained in a tuff sample collected from the Heitaro-zawa Formation. A, cathodoluminescence image (CL) of zircon grains from the sample. Circles on the grains represent spots analyzed by LA-ICP-MS. Spots are 25 μm across. B, Tera-Wasserburg U–Pb concordia diagram of zircons. C, ^{238}U–^{206}Pb age distribution plot of zircon grains in the tuff sample. $^{207}Pb^*$ and $^{206}Pb^*$ indicate radiometric ^{207}Pb and ^{206}Pb, respectively.

the samples show rhythmic oscillatory and/or sector zoning on cathodoluminescence images, which is commonly observed in igneous zircons (e.g. Corfu *et al.*, 2003), and their higher Th/U ratios (>0.1) also support their igneous origin (Williams and Claesson, 1987; Schiøtte *et al.*, 1988; Kinny *et al.*, 1990; Hoskin and Black, 2000). Figure 2 shows a Tera-Wasserberg concordia diagram and an age distribution plot for all analyzed spots of the sample. Data are obtained from 28 spots, of which data for 27 are concordant. U–Pb ages cluster in the range 66.8–73.7 Ma. The weighted mean age of data for 25 spots, excluding data for 3 spots (2 inheritance and 1 discordant), yield 69.8 ± 0.8 Ma (MSWD = 1.2; 95% conf.), which is thought to represent the magmatism/deposition age of the tuff sample.

Discussion

Based on the magnetostratigraphy of the Senposhi Formation in eastern Hokkaido, Shigeta *et al.* (2015) assigned the portion of the *Pachydiscus flexuosus* Zone (taxon-range zone) that contain *Gaudryceras makarovense* Shigeta and Maeda (Maeda *et al.*, 2005) in the lower part, to the middle to upper part of the middle Maastrichtian. However, since

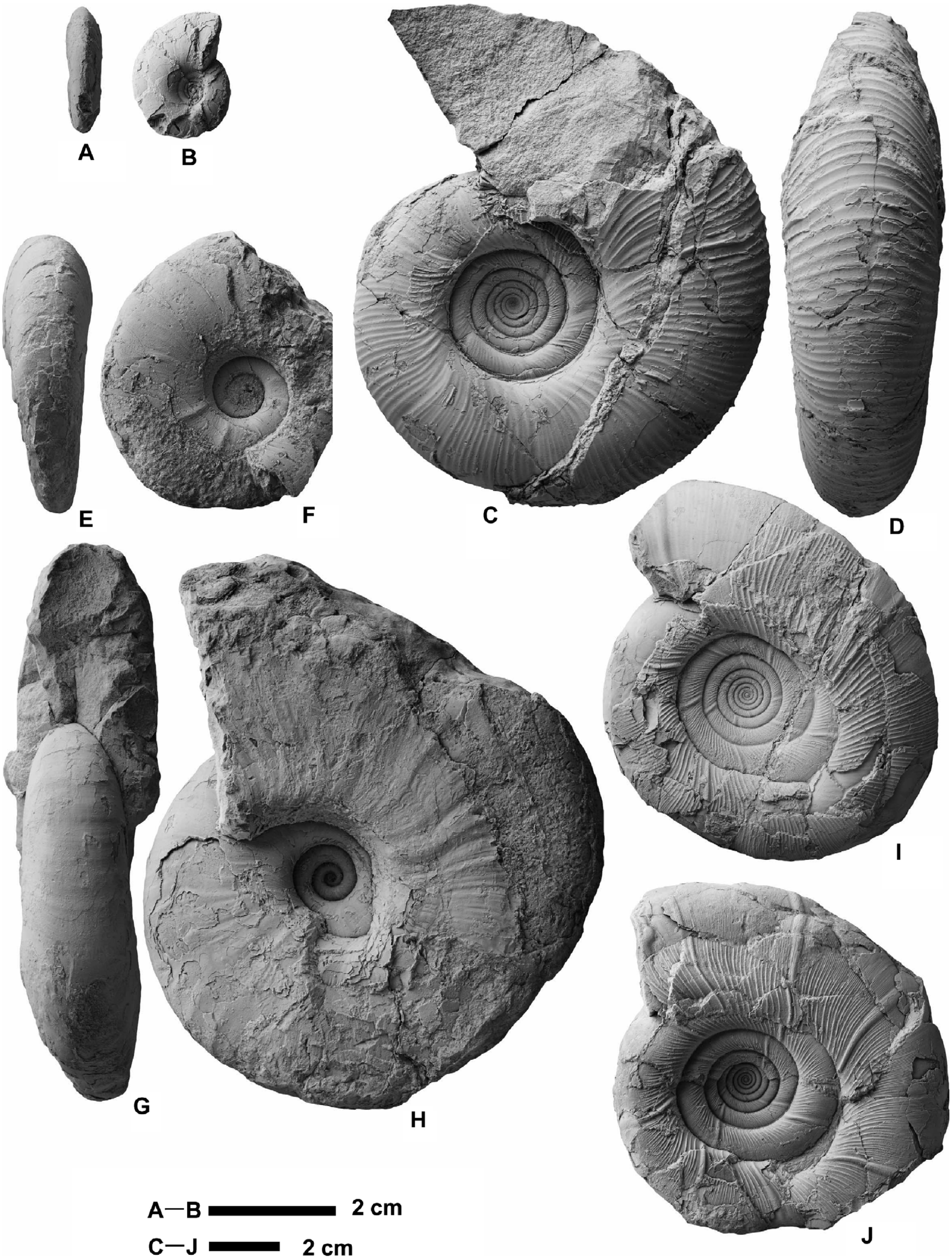

Fig. 3. Important ammonoids from the Heitaro-zawa Formation along the Nakanokawa River. A, B, *Zalandites varuna*, NMNS PM35456, from loc. 1036; C, D, *Gaudryceras makarovense*, NMNS PM35457, from a float concretion found at loc. 2032; E–H, *Pachydiscus flexuosus*; E, F, NMNS PM35458, from loc. 2033 (collected by Y. Tsujino); G, H, NMNS PM35459, from loc. 2034; I, *Gaudryceras* sp., NMNS PM35460, from a float concretion found at loc. 2035; J, *Gaudryceras hamanakense*, NMNS PM35461, from loc. 2037.

the lowermost portion of the Senposhi Formation and the underlying formation are unfossiliferous, the lower limit of the stratigraphic distribution of *P. flexuosus* and its age are not yet known. Because *G. makarovense* was collected from a float concretion that most likely came from loc. 2032 (Fig. 1), the *P. flexuosus*-bearing beds at locs. 2034 and 2033 underlie the *G. makarovense*-bearing beds (Figs. 1, 3). Zircon geochronology in this study reveals that the U–Pb age of the tuff bed at loc. 2034 is

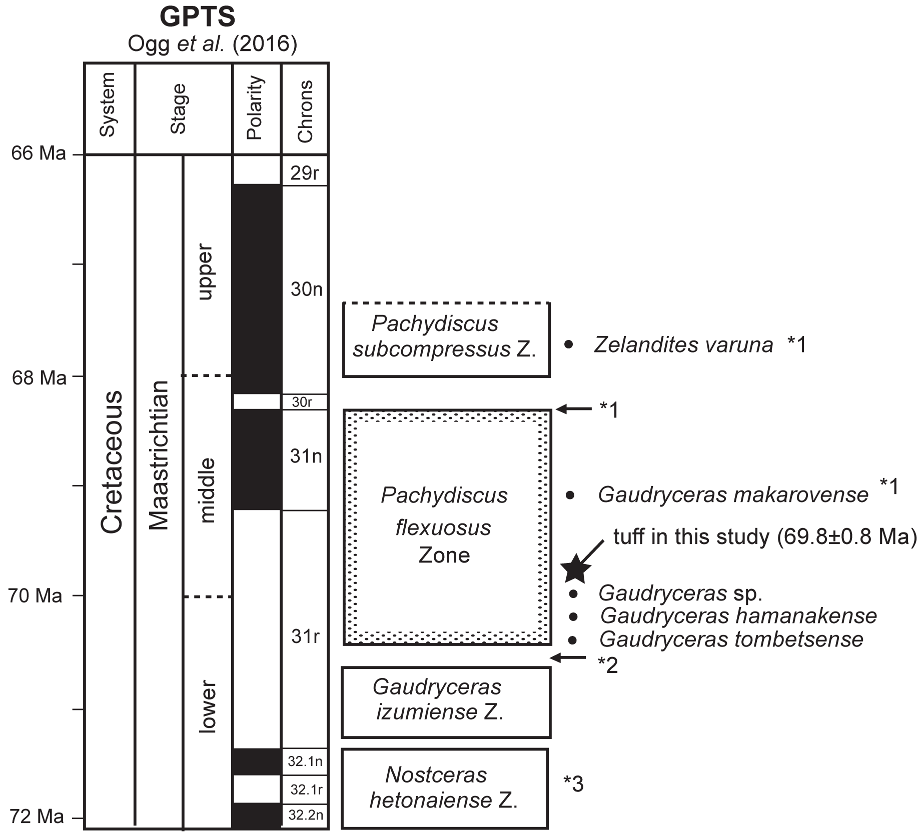

Fig. 4. Diagram showing Maastrichtian ammonoid zones (taxon-range zones) and selected ammonoids in the Northwest Pacific region, based on new data presented in this paper, Shigeta *et al.* (2015: *1), Shigeta *et al.* (2017: *2) and Kodama (1990: *3).

69.8 ± 0.8 Ma (95% conf.), which corresponds to a late early to early middle Maastrichtian age, suggesting that the *P. flexuosus* Zone at least spans the entire middle Maastrichtian.

In the Nakanokawa River section of the Nakatonbetsu area, *Gaudryceras hamanakense* Matsumoto and Yosida, 1979 occurs at loc. 2037 and *Gaudryceras* sp. was collected from a float concretion that most likely came from loc. 2035. With regard to ornamentation, the ribs of *Gaudryceras* sp. are coarser than those of *G. hamanakense* and finer than those of *G. makarovense*. Based on this observation, a specimen attributed to *G. makarovense* at MK2023 from southern Sakhalin by Maeda *et al.* (2005, figs. 32.4, 32.5) is identical to *Gaudryceras* sp. Because *Pachydiscus flexuosus* co-occurs with *G. hamanakense* in eastern Hokkaido (Naruse *et al.*, 2000) and southern Sakhalin (Maeda *et al.*, 2005), it is obvious that the tuff bed at loc. 2034 does not represent the bottom of the *P. flexuosus* Zone. The exact age of the *Gaudryceras hamanakense*-bearing beds has not yet been studied, but Shigeta *et al.* (2017) have determined the U–Pb age of a tuff bed overlying the *Gaudryceras izumiense*-bearing beds, which underlie the *Pachydiscus flexuosus* Zone in Hokkaido (Toshimitsu *et al.*, 1995), to be 70.5 ± 1.1 Ma (95% conf.) (= late early Maastrichtian). Maeda *et al.* (2005) illustrated a specimen they attributed to *P. gracilis* Matsumoto (Matsumoto *et al.*, 1979) from the *G. tombetsense*-bearing bed in the Makarov area, but Shigeta *et al.* (2012) later referred it to a peculiar morphotype of *P. flexuosus* that exhibits a much more compressed whorl section. The *G. tombetsense*-bearing beds are located below the *G. hamanakense*-bearing beds in the Makarov area (Maeda *et al.*, 2005). These facts suggest that the lower part of the *P. flexuosus* Zone containing *G. hamanakense*, or *G. tombetsense* just below it, is probably equivalent to the upper lower Maastrichtian. (Fig. 4).

As indicated earlier by Shigeta *et al.* (2010, 2012) and Shigeta and Nishimura (2013), *Gaudryceras* is an ideal ammonoid genus for precise biostratigraphic correlation of the Maastrichtian in the Northwest Pacific region, e.g. *Gaudryceras tombetsense*, *G. hamanakense*, *Gaudryceras* sp. and *G.*

makarovense occur in ascending stratigraphic order in the *Pachydiscus flexuosus* Zone in the Makarov area, southern Sakhalin (Maeda *et al.*, 2005; Shigeta *et al.*, 2012). Future taxonomic and biostratigraphic studies of these *Gaudryceras* taxa will more accurately define the subdivision of the *P. flexuosus* Zone and provide an important key for the establishment of a precise biostratigraphic framework for the Maastrichtian in the Northwest Pacific region.

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