Symmetry of iridescent garnets from Tenkawa, Nara Prefecture, Japan

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Grossular (Ca3Al2Si3O12) - andradite (Ca3Fe3+2Si3O12) garnet solid solutions (space group, Ia-3d), termed grandite series, often exhibit optical birefringence and iridescence. Iridescent garnets (rainbow garnet) is a variety name for the iridescent Fe3+-rich grandite. Shimobayashi et. al (2005) examined the iridescent garnets from Tenkawa, Nara Prefecture, Japan and they reported the presence of fine lamellar texture (ca. 100-300 nm in thickness) with small differences in chemical composition (Al/Fe3+ ratio) by transmission electron microscope (TEM). They also suggested that Al-rich fine lamella should be reduced in symmetry from cubic system, but they did not show the direct evidence of the symmetry reduction. Our previous study (Chang et al., 2016) reported that extra reflections, 110, 200, 411 and so on, to break the symmetry of Ia-3d were detected, in selected area electron diffraction (SAD) patterns from Al-rich fine lamellae. In this study, we examine convergent-beam electron diffraction (CBED) method in addition to SAD method by TEM to investigate the symmetry of same iridescent garnet from Tenkawa.

A thin section (100μm thickness) cut parallel to the (001) face through the center of the crystal was prepared from an euhedral single crystal of iridescent garnet with well-developed rhombic dodecahedral {110} facets. TEM specimens with various crystal planes, (001), (1-10), and (111) were prepared from the thin section by using a focused ion beam technique (FIB, FEI: Quanta 200 3DS) and then TEM (JEOL JEM-2100F) observation was carried out to investigate the symmetry.

SAD patterns obtained from Fe-rich fine lamellae reveals that the Ia-3d symmetry retains as the usual garnet and extra reflections did not appear, although 200 and 020 reflections appear for multiple (double) diffraction. On the other hand, in SAD patterns from Al-rich fine lamellae, extra reflections, 110, 200, 411 and so on, to break the symmetry of space group (Ia-3d) were detected, indicating that a-glide and d-glide planes should be lost. However, all reflections satisfy the reflection condition of body centered lattice (I-lattice, hkl; h+k+l=even). Furthermore, the each intensities of 110, -110, -1-10 and 1-10 have different counts and this indicate that symmetry in this area does not have fourfold rotation axis. CBED pattern in (111) TEM sample indicates no threefold rotation axis and CBED in (001) sample shows no mirror planes perpendicular to a- and b-axes and no twofold rotation angle parallel to a-, b- and c-axes. Moreover, CBED in (1-10) also shows no mirror planes perpendicular to c-axis. These results indicate crystal family of this Al-rich fine lamella is triclinic and lattice type is I-lattice and suggest the space group is I-1 or I1.

Reference

Shimobayashi et al. (2005) Abstracts of Annual Meeting of the Mineralogical Society of Japan ,p80.
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Keywords: iridescent garnet, TEM, SAD and CBED

Coloration Analysis of Yellow Synthetic Diamonds

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Almost all of the high temperature-high pressure (HTHP) synthetic diamonds have yellow tone, which is discussed and solved in this thesis. The yellow synthetic diamond samples are tested with UV –visible light spectrophotometer, micro-infrared spectroscopy, cathode luminescence and other large equipments. We focus on high temperature and high pressure conditions changing, and the following conclusions are made :In the high temperature under vacuum pressure, diamond is not in the stable phase region, graphite can easily occur. Under experimental high temperature and pressure conditions (1500-1600 ℃, 7.50GPa), the color of samples in this thesis have changed, the yellow tone has been obviously decreased, and the experiment is repeatable. Micro-infrared spectroscopy reveals, significant peak intensity and position changes occurred in 1100-1500cm^{-1} spectral region of nitrogen. UV - visible spectrophotometer test of the samples before and after experiment shows that the color has changed, the samples become light-colored. Synthetic diamonds at HTHP treatment changes occurred in diamond type from IaA + Ib to IaAB + Ib. The band theory can explain the reasons for diamond’ s coloration.

Keywords: Synthetic diamond, HTHP, Coloration

Ghost zones in sediment-hosted copper deposits

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It has been said that a mineral zonation in a sediment-hosted copper deposit appears as, from an upstream side, chalcocite - bornite - chalcopyrite - pyrite. However, the well-developed zonation has rarely been reported in the previous investigations in the deposits. In many deposits, wide monomineralic chalcocite and/or native copper zones have been observed, while chalcopyrite and/or bornite could be seen in narrow areas vicinity to pyrite zones.

Although several formation models of the deposits have been proposed, it is widely believed that the copper mineralization was caused by reducing copper bearing oxic fluids during their migration in sediments or sedimentary rocks. Therefore, the formation processes of the zonation may be investigated by analytical schemes for processes of reactive transport (infiltration metasomatism).

Let us suppose that mineral B is stable between minerals A and C, that is, the assemblage A + C is incompatible due to B. Minerals A, B and C are comparable with chalcocite, bornite and chalcopyrite, respectively, in the above deposits. Then, we may think the zoning as, for example, from the upstream side, A - B - C. However, we need to confirm if the velocity of the replacement front of C by B is surely faster than that of B by A. If not, the zone B becomes as a ghost zone, that is, C is directly replaced by A, while the solution at the front is in equilibrium with B and C, since C can not equilibrate with A. The practical meaning may be that C is replaced by B being replaced by A soon after its formation. Therefore, the zone B does not expand with time.

Analytical solutions of the velocities of the replacement fronts in the above mineral zonation in sediment-hosted copper deposits have been obtained by Mathematica (Wolfram Research), while the formation processes have been numerically simulated by MIX99 (Hoshino et al., 2000). The results are:

(1) Although primary reductants may be carbonaceous matters in sediment (rock), the front velocity of pyrite precipitation due to reducing fluids by carbonaceous matters is quite fast, so the direct reductant for the following fluids is pyrite.

(2) Chalcocite monomineralic zone is formed after (upstream side of) the pyrite dissolution front when the inlet fluid pH is low.

(3) Native copper zone and/or chalcocite zone are formed (in this sequence) after the pyrite dissolution front when the pH is high.

(4) Bornite and chalcopyrite do not form the zones in all analyzed conditions, but ghost ones vicinity to the pyrite dissolution front when they appear.

It can be concluded from the analytical results that bornite and chalcopyrite are transitional minerals even when they precipitate during mineralization, so that their well-developed zones have rarely be seen in the sediment-hosted copper deposits.

Keywords: sediment-hosted copper deposit, mineral zonation, ghost zone

A Novel Analysis Framework for Particle Morphological Classification of Mineral Ore Using A Statistical Raman Analysis and X-ray Diffraction Analysis.

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[Introduction]

Mineral ores extracted by mining go through a milling process before ore dressing. An important factor in both milling and ore dressing operations is the determination of the particle size distribution of the materials being processed, commonly referred to as particle size analysis. An elemental analysis technique such as X- ray based analytic technics and destructive wet chemical analysis can determine the quantity of mineral species present in the ore. Especially, X ray diffraction analysis (XRD) is commonly used as non-destructive and high sensitive analysis to investigate of a component analysis of minerals in ore based on the assumption methodologies such as Rietveld quantitative analysis. However, these existing chemical and elemental analysis methods do not allow the study of the composition of individual particles of different size and shape. Our group had been reported for capability of the statistical Raman spectroscopic method (SRS) is novel approach which can resolve this problem [1]. Using this method the Raman spectra of several hundred particles is determined after size and shape classification of each individual particle by an automated particle image analysis (APIA). Raman spectroscopy can be used to acquire the spectra of any inorganic compounds such as metal oxides and nitrides which are Raman active. Many mineral resources are mined as inorganic compounds. Therefore, Raman spectroscopy can be used for the identification of the chemical composition of mineral ores. Using SRS method described herein, it is possible to calculate the particle size distribution and proportion by mass or volume of each chemical component or mineral species based on Raman spectroscopic information. However, most of drawback of SRS method is not allow to determine for the absolute vale of component in mineral ore, therefore, XRD is needed as complementally method. This study will report and discuss the capability about a combined novel analytical framework of SRS method and XRD using iron ore as a model material.

[Material and Method]

Iron ore samples were purchased from a vendor. These samples had been through the ore dressing process. XRD was carried out using an Empyrean (PANalytical, Netherland) equipped Cu Kα X ray source, performed over a range of $20^\circ < 2\theta < 100^\circ$, with a step size was 0.026. To make sure averaging sample information due to a powder segregation, a sample spinner was used. SRS analysis was carried out using a Morphologi G3-ID instrument (Malvern Instruments, UK) equipped with a dry powder sample dispersion unit (SDU) and Raman module. The laser wavelength of Raman excitation was 785nm the laser power was less than 5mW. Iron ore dry powder samples were dispersed using the SDU using a short duration pulse of compressed air. Measurements were made automatically using Standard Operating Procedures (SOPs) which define the software and hardware settings used. Measurement sample was dispersed on to glass plate as sample carrier which was minimized environmental exposure by the enclosed sample chamber unit. Particle identification by Raman analysis used the spectrum correlation coefficient approach.

[Results and Discussion]

A qualitative analysis by XRD profile had suggested for that sample were including of a polymorphic Iron oxides such as Fe_2O_3, F_3O, Fe_3O_4 and hydroxide $F_3O(OH)$. Population of component were Iron Oxide 93.7%, Magnetite 1.5% , Goethite 1.7% and Quartz 3.1% were investigated which were calculated based on Rietveld quantitative analysis. Raman spectrum SRS analysis was performed to investigate of the

population analysis on several size fractionated based on Raman spectrum classification and APIA analysis.

[Summary]

This report illustrated the application and capability of a combined novel analytical framework of SRS method and XRD.

[1] Sasakura D, Hayauchi A., “A Novel Approach for the Classification of Mineral Ore Particles by A Statistical Raman Spectroscopic Method” ., SCG-6-11, JpGu Annual Conference 2017, Yokohama,

Keywords: XRD, Raman Spectroscopy, Particle Size and Shape

Weathering process of basalt with cooling joints in the Takurayama volcano in the northern Kinki region, Japan

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Columnar joints and low-angle joints are well-developed in basaltic lava layers of the Quaternary Takurayama Volcano in the northern Kinki, Japan. The basaltic columns are separated into smaller prisms with average sides of 0.4 m long and heights of 0.1-1 m; the prims are weathered to become spherical corestones with scale-like rindlets. This weathering style is referred to as spheroidal weathering. Mineralogical and chemical processes of spheroidal weathering have been studied in many types of igneous rocks but the effects of original rock structure on the weathering have not been explored sufficiently. Consequently, the whole mechanism of spheroidal weathering has not been elucidated enough. We conducted a field survey of cooling joints, measured pore-size distribution in rock specimens, and analyzed their structures, chemistry, and mineralogy. We found there are internal cracks that trim the corners of joints, and concentric dark layers in columns like yearly growth rings, which were the layers with smaller pores: These original rock structures encourage the spheroidal weathering. Weathering of olivine and plagioclase advances inward from joint surfaces. Water from the joint surfaces advances inward, oxidizing olivine to form iddingsite and iron-hydroxides and transforming plagioclase to halloysite, which then is dissolved to leave gibbsite at the joint surfaces. The inward advancement of the weathering is retarded by the dark bands. Thus, weathered layers including halloysite develop with a concentric shape and then exfoliate one after another, forming spheroidal core stones.

Keywords: spheroidal weathering, columnar jointing, basaltic lava, pore-size distribution

Thermal history of lithospheric mantle: Mantle xenolith from Colorado Plateau in North America continent revisited

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Mantle convection plays an important role in heat and mass transportation from the deep earth to the surface. It is thus important to understand the thermal state of the mantle and its temporal changes, direct information of which may be obtained from fragments of the mantle brought to the surfeace by magmas, usually called “mantle xenoliths”. We study the mantle xenolith from continental lithosphere to reveal dynamics of chemical and thermal interaction taking place near the lithosphere asthenosphere boundary in the continental region. The target area is the Thumb in the Navajo volcanic field in the Colorado Plateau of the North America continent (Ehrenberg,1982). Five xenolith samples from the Thumb were thoroughly examined by using optical microscope, electron probe micro analyzer (EPMA), and field-emission secondary election microscope(FE-SEM). Our strategy is quantitative comparison of microstructures and mineral chemical compositions among examined xenolith to reveal their depth variation of thermal, chemical, and rheological properties

Examined five samples are two garnet lherzolite, garnet harzburgite, without fine-grained portions, and the other contains fine-grained recrystallized olivine in various amounts. Previous study (Ehrenberg,1982) argued that the mantle xenolith from the Thumb are equilibrated at various temperature with almost no systematic variation in the depth of xenolith derivation. The five samples analyzed this study show a wide variation in CaO contents in olivine from 0.02˜0.08 wt.%. A garnet lherzolite sample with lower CaO contents in olivine (0.03˜0.04 wt.%) contains clinopyroxene and orthopyroxene with strong zoning characterized by enrichment of Al and Ca towards the rim. Garnet grains in the sample shows extensive decomposition via reaction with olivine into aggregates consisting of spinel and pyroxenes with diverse size change: coarse in the outer margin (˜several tens of micrometer) and very fine near the contact with relict intact garnet (˜1 micrometer). The pyroxene zoning and breakdown texture of garnet suggest that the xenolith underwent heating or decompression before this mantle xenolith was brought to the surface by the host minett. Preliminary geothermobarometry has revealed pressure-temperature correlation, from which the xenoliths records the temperature variation of ˜200℃ over the pressure range of ˜25GPa. The estimated pressure correlates with various textural features, such as size of recrystallized olivine, which decreases with depth . They also show correlations with mineral chemical parameters, such as forsterite content of olivine, which increases with depth, suggesting that the lithosphere is more depleted with depth .

Keywords: mantle xenolith, thermal history of lithosphere

Petrogenesis of basalts from Emeishan large igneous province constrained from major element, trace element and Pb isotope compositions of olivine-hosted melt inclusions

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Large igneous provinces (LIPs) are produced by some of the largest known volcanic episodes on our planet. The Emeishan LIP in SW China is one of the most significant LIPs in the world as it comprises several world-class giant V-Ti magnetite deposits and possesses some Cu-Ni-(PGE) sulphide deposits. In addition, the Emeishan LIP is thought to be one of the best examples of a LIP generated by a mantle plume. Furthermore, the Emeishan volcanism might have caused the end-Guadalupian mass extinction. In the last few decades, numerous petrological, geochemical, paleontological, paleomagnetic, geophysical, geochronological, and mineral deposit studies have been conducted on the Emeishan LIP. Despite the breadth of understanding, there are still many controversies concerning the compositions of the primary magmas, the lithology of their sources, and the melting processes in the sources. It has long been believed that the source of the Emeishan basalts is peridotite, either from a mantle plume or the sub-continental lithosphere. Olivine-hosted melt inclusions within lava retain information regarding the lava’s primary magma compositions and mantle sources. Thus, they can be used to infer the nature of the mantle sources of large igneous provinces, which is still not well known and of the subject of debate. We have analyzed the chemical compositions and Pb isotopic ratios of olivine-hosted melt inclusions in the Dali picrites, Emeishan Large Igneous Province (LIP), SW China. These are the first *in-situ* Pb isotope data measured for melt inclusions found in the Emeishan picrites and allow new constraints to be placed on the source lithology of the Emeishan LIP. The melt inclusions show chemical compositional variations, spanning low-, intermediate- and high-Ti compositions, while their host whole rocks are restricted to the intermediate-Ti compositions. Together with the relatively constant Pb isotope ratios of the melt inclusions, the compositional variations suggest that the low-, intermediate- and high-Ti melts were derived from compositionally similar sources. The geochemical characteristics of melt inclusions, their host olivines, and whole-rocks from the Emeishan LIP indicate that Ca, Al, Mn, Yb, and Lu behave compatibly, and Ti, Rb, Sr, Zr, and Nb behave incompatibly during partial melting, requiring a pyroxenite source for the Emeishin LIP. The wide range of Ti contents in the melt inclusions and whole-rocks of the Emeishan basalts reflects different degrees of partial melting in the pyroxenite source at different depths in the melting column. The Pb isotope compositions of the melt inclusions and the OIB-like trace element compositions of the Emeishan basalts imply that mixing of a recycled ancient oceanic crust (EM1-like) component with a peridotite component from the lower mantle (FOZO-like component) could have underwent solid-state reaction, producing a secondary pyroxenite source that was subsequently partially melted to form the basalts. This new model of pyroxenite melting could explain the geochemical variations among the low-, intermediate- and high-Ti basalts for the Emeishan LIP and challenges the prevailing belief that the source of the Emeishan basalts is peridotite.

Keywords: large igneous province, pyroxenite source, Pb isotope of melt inclusion, olivine-hosted melt inclusion, mantle plume

Disequilibrium melting and assimilation of granitic xenoliths in andesitic magmas

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Crustal assimilation is one of the important magmatic processes especially for arc magmas. This process is well-illustrated at Mt. Tsukumo in NE Shikoku, SW Japan. Mt. Tsukumo is a small volcanic neck composed of Miocene andesites (Setouchi volcanic rocks), which locally contains partially melted granitic xenoliths (10–60 cm). By using the samples, we examined the petrological and geochemical evolution during xenolith assimilation on sub-meter scale.

A suite of rock samples indicates that xenolith assimilation proceeds through disequilibrium melting of granitic xenoliths and subsequent mixing of xenolith-derived porphyritic magmas with host andesitic magmas. Minerals in xenoliths are separated along phase boundary by melting, and further broken into smaller pieces by injection of partial melt into thin fractures. These minerals except for quartz progressively change their texture and composition through dissolution and precipitation. The partial melts in xenoliths, now observed as fresh glass, show diffusion-controlled compositional variations, and its heterogeneity is significant in Al_2O_3 and SiO_2. The heterogeneity of melt remains even under moderate degree of melting (~40%). Observation suggests that partially melted xenoliths behave as magma, not rigid material, when the melt proportion exceeds 50 vol%. Thus, the xenolith-derived magmas mix with host andesitic magmas to form a mixing zone surrounding xenoliths. This mixing event erases the diffusion-controlled compositional heterogeneity of partial melts. Consequently, compositions of assimilated magmas are approximated by binary mixing of the host andesitic magmas with the xenolith-derived magmas that experienced >50 % melting.

The Setouchi volcanic rocks, including high-Mg andesites, often contain quartz and feldspar xenocrysts, which are probably from Cretaceous granitic basement. This study suggests that the volume of granitic xenolith-derived melts cryptically mixed with host magmas is more than double of the observed xenocryst abundance. In addition, occurrence of quartz xenocryst should be investigated whether they were derived from partially-melted xenoliths or non-melted xenoliths. Quartz is easily separated from other granite-forming minerals at the early stage of partial melting. Thus, isolated quartz xenocrysts dispersed in host magmas could be derived from partially-melted xenoliths. If quartz form an aggregate with other mineral phases such as feldspar and biotite, these minerals would be entrained by mechanical breakoff of non-melted xenoliths.

Keywords: xenolith, assimilation, disequilibrium melting

Variation of the average aspect ratio of plagioclase in the Atsumi dolerite sills, Northern Japan

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To clarify the time scale of cooling and crystallization of magma in igneous rocks is important for determining the time scale of magma processes. According to experimental studies of plagioclase, it is shown that increasing undercoolings and cooling rates result in greater differences between the growth rates of the different crystallographic faces, resulting in grains which are elongate parallel to [100] (Muncill and Lasaga, 1988; Pupier et al., 2008). Recently, Holness (2014) found that there is a good correlation between the average aspect ratio and the crystallization time in six sills which have relatively similar compositions and different thicknesses and showed that the aspect ratio is useful as a speedometer in igneous intrusive bodies. In this paper, it is concluded that the shape of plagioclase is relatively insensitive to small variations of magma composition, but the influence of magma composition on the aspect ratio is not discussed in much detail. Therefore, in order to examine the influence of magma composition on the aspect ratio, we conducted the measurement of the plagioclase aspect ratio and the calculation of the crystallization time for two alkaline basaltic sills in the Atsumi district (Kayaoka sheet, 120 m in thick; an intrusive body in Hakuzan island, 6 m in thick; Kushiro 1964).

As a result, the following results were obtained for plagioclase from the two sills: (1) The ranges of the average aspect ratios (major axis length / minor axis length) are about 3.8-5.6 and 6.4-9.8 for the Kayaoka sheet and the intrusive body in Hakuzan island, respectively. (2) There is a negative correlation between the average aspect ratios and the calculated crystallization times in the two sills. (3) In the Kayaoka sheet, the average aspect ratio increases from the central part towards the top and bottom but decreases again only in the undermost few meters (Fig.1; S-shaped profile). The features of (2) and (3) are the same as those reported in Holness (2014). However, compared between sills of comparable thickness, average aspect ratios in our study are significantly larger than those in Holness (2014) (for example, the average aspect ratio in the 129-meter-thick Whin sill is about 3.0 - 3.6). The differences in plagioclase aspect ratio are likely to be caused by the influence of magma viscosity on diffusion coefficient.

Keywords: aspect ratio, cooling rate, Atsumi dolerite

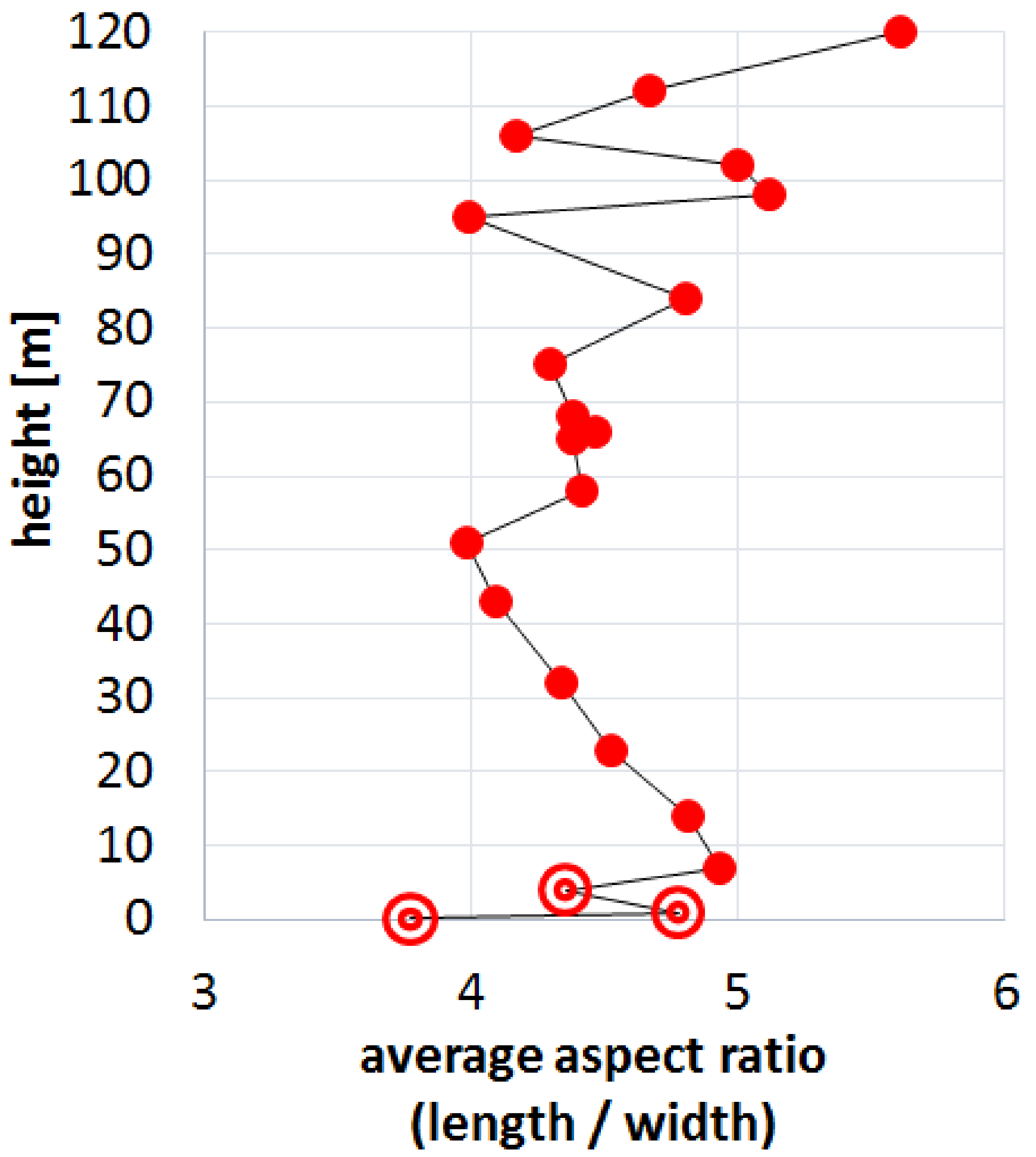

Fig.1 Stratigraphic variation of plagioclase average aspect ratio in the Kayaoka dolerite sheet.

Petrochemistry and U-Pb zircon geochronology of felsic rocks from the Kagura complex, south Kitakami Mauntains, Japan

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Basement rocks of the South Kitakami belt are distributed in Hayachine to Miyamori area, and is called Hayachine complex. Petrochemistry and U-Pb zircon geochronology were investigated from felsic rocks in the Kagura complex associated with Hayachine complex. U-Pb dating of zircons were carried out using Agilent 7500cx quadrupole inductively coupled plasma mass spectrometer (ICP-MS) with a New Wave Research UP-213 Nd-YAG UV (213 nm) laser ablation system (LA) installed at the Kyushu University (Adachi et al., 2012). Zircon grains from the tonalite define a concordant age of 494+/-4Ma (n=18) and 487+/-4Ma (n=26), and from the quartz diorite define a concordant age of 500+/-4Ma (n=19). These data suggests that age of the Hayachine complex could become older ca. 500Ma. Moreover, granitic activity in early Paleozoic in proto-Japan occurs at ca. 500 Ma and ca. 450 Ma.

Keywords: zircon geochronology, Kitakami Mountains, Cambrian