Ages of Zircons in Jadeitite and Jadeite-bearing Rocks of Japanese Islands

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Abstract Radiometric ages of zircons in jadeitite and jadeite-bearing rocks from five areas in Japan were obtained from the $^{238}\text{U}/^{206}\text{Pb}$ ratio using a sensitive high resolution ion microprobe (SHRIMP II). The samples exist as blocks in serpentinite or greenstone mélangé from Itoigawa (IT), Osayama (OS), Kochi (KC), Nishisonogi (NS) and Yorii (YR) areas. Coarse-grained zircon from IT yields $^{206}\text{Pb}/^{238}\text{U}$ mean age of 497±23 Ma and two zircons from the OS yield different mean ages; 496±12 Ma and 532±17 Ma, respectively. Fine-grained zircons in the jadeite rocks from KC, NS and YR yield mean ages; 501±5 Ma, 136±5 Ma and 159±5 Ma, respectively. The protolith of the KC sample is considered to be a tectonic block which was a crustal material formed ~500 Ma and once subducted to certain depth of jadeite formation after tectonic erosion of the crust. The zircon age of the IT sample are consistent with previously reported values which is interpreted as an age of jadeitization. Age difference of two zircon grains from OS sample shows a possibility that zircons were not formed by a single event but multiple events. Zircons in the NS and YR samples are interpreted as protolith or hydrothermal origin. It is clear that the zircon ages of jadeite rocks are roughly bimodal; middle Mesozoic (NS and YR) and early Paleozoic (IT, OS and KC).

Key words: zircon, radiometric age, jadeite rock, origin

Introduction

Jadeitite and jadeite-bearing rocks are found at restricted number of localities in the world. Most of them exist as blocks in serpentinite or greenstone mélangé associated with high-P/T type metamorphic rocks, but there are some varieties of jadeite situation; “dyke” or pod in serpentinite, vein in high-P/T type schist (reviewed in Harlow and Sorensen, 2005). In Japan, although gem quality jadeitite is found only at the Itoigawa area, there are more than ten localities of jadeitite and jadeite-bearing rocks (Fig. 1). Jadeites at the Mikkabi and Yatsushiro areas occur as a vein cutting through metagabbro (Yokoyama and Samejima, 1982; Saito and Miyazaki, 2006). Jadeite-bearing rock from the Shimonita area shows pillow texture and, therefore, is considered to be derived parentally from oceanic basalt (Tanabe et al., 1982). Jadeite-bearing rock from Kamikutotan is a mafic rock consisting mostly of amphibole (Gouchi, 1983).

Jadeite rocks often contain zircon as accessory mineral and the zircon U–Pb ages are interpret as several mineralization stages; protolith or pre-jadeitization age, jadeitization age, or age of late stage hydrothermal process (e.g. Shi et al., 2008). In this study, zircons were found in the jadeitite and jadeite-bearing rocks from the five areas which are tectonic blocks in serpentinite or greenstone mélangé: Itoigawa, Osayama, Nishisonogi, Kochi and Yorii areas, and U–Pb ages of zircons from these five areas were obtained using an ion microprobes. Although zircon
ages of jadeite rocks do not have direct relation to the timing of formation of mélangé including the jadeite rocks, the ages would have some information about the framework of the Japanese Islands.

Geological Settings

Itoigawa area, Hida Mountains (IT)

This area is assigned to the Hida Gaien Belt which is a serpentinite mélangé zone with various type of tectonic blocks; high-P/T type metamorphic rocks, weakly metamorphosed sedimentary rocks, amphibolites, metamorphosed mafic rocks, jadeitites, albitites and rodingites (Nakamizu et al., 1989). K–Ar, 40Ar/39Ar and Rb–Sr ages were obtained from high-P/T type metamorphic rocks of around 300 Ma (Shibata and Nozawa, 1968; Shibata et al., 1970; 1979) and eclogites of 340 Ma (Tsujimori et al., 2001). The weakly metamorphosed sedimentary rocks are Carboniferous to Permian from fossil evidence (Tazawa et al., 1984). Nishimura (1998) suggested the Hida Gaien Belt is a part of the Renge Belt. We selected a lavender color jadeitite from the stored specimen in National Museum of Nature and Science (hereafter NMNS) (register number: NSM-M-29604) which collected from Kotakigawa River (around N36°55’ E137°48’) as a boulder. The sample consists almost completely of jadeite, with small amounts of natrolite, zircon, titanite, tautsonite and rengeite. Zircon in the sample is subhedral chasmy crystal with few millimeters in diameter. The ion microprobes U–Pb age of zircons in two types of jadeite rock from this area were reported as $519^{+17}_{-16}$ Ma and $512.3^{+6.9}_{-7.0}$ Ma (Kunugiza et al., 2002).

Osayama area, Chugoku Mountains (OS)

The Osayama area is a serpentinite mélangé which contains small bodies of rodingite, albitite, jadeitite, and tectonic blocks of high-P/T type metamorphic rocks (Kobayashi et al., 1987; Tsujimori, 1997). Phengite K–Ar ages of the metamorphic rocks yield 289–327 Ma and they concentrated around 320 Ma which are comparable to the age of Renge Belt (Tsujimori and Itaya, 1999). U–Pb age of zircon in jadeitite from this area was reported as $472.0^{+8.5}_{-8.1}$ Ma (Tsujimori et al., 2005). Two zircon grains were separated from jadeitite which is collected from the same outcrop as the jadeitite studied by Tsujimori, et al. (2005). They are a few millimeters in diameter and are subhedral clear crystals. The samples are the stored specimen in NMNS (register number: NSM-M-24785).
Kochi area (KC)

The Kochi area belongs to the Kurosegawa Belt which is a serpentinite mélange with various rock types of tectonic blocks; Silurian and Devonian sediments, granites, gneisses, amphibolites, basic schists and pelitic schists (e.g. Ichikawa et al., 1956). K–Ar ages of muscovite in jadeite-bearing schist were 208–240 Ma (Maruyama et al., 1978). The jadeite rock studied here is found as a boulder at Funakoshi, Hidaka Village, Kochi Prefecture (around N33°/H11034°32’/H11032°; E133°20’). The locality is 15 km WSW away from the famous locality of Engyoji area, eastern extension of serpentinite mélange of Kurosegawa Belt. The sample consists mainly of jadeite and quartz with a subordinate amount of glaucophane. The sample is the stored specimen in NMNS (register number: NSM-R-117953). Zircon in this sample is euhedral with length of 100 to 300 µm.

Nishisonogi area (NS)

The sample of the Nishisonogi area was collected as a boulder at Tone, Nishisonogi Peninsula (N32°53.5’/E129°45.8’) and jadeite rock of the area was reported by Shigeno et al. (2005). The area belongs to the Nagasaki Metamorphic Belt with K–Ar age of 60–90 Ma (Hattori and Shibata, 1982). The metamorphic belt consists mainly of pelitic, psammitic and basic schists with a small amount of serpentinite. The serpentinite occurs as a small pod or serpentinite mélange containing various types of tectonic blocks; metabasites, amphibolites, jadeitites, omphacite rocks, rodingites, zoisite rocks and epidote–garnet–crossite–barroisite rocks (Nishiyama et al., 1989). The major component of the jadeite rock is jadeite and albite; content of jadeite is 65–93%. Quartz is rarely observed as an inclusion in jadeite (Shigeno et al., 2005). The sample of jadeite rock is a stored specimen in NMNS (register number: NSM-R-117953). Zircon in this sample is euhedral with length of 100 to 300 microns.

Yorii area (YR)

The jadeite-bearing rock of the Yorii area is found as a huge block ca. 30 m in length associated with greenstone and serpentinite developed at the boundary between the Cretaceous Tochiya Formation and the Sanbagawa high-pressure type metamorphic belt (Hirajima, 1983). It consists mainly of jadeite, quartz and albite with a small amount of glaucophane. Jadeite is highly replaced by albite. Jadeite content is up to 50%. The Sanbagawa metamorphic rock has K–Ar age around 60–90 Ma (Miyashita and Itaya, 2002; Hirajima et al., 1992). No age datum was reported from the jadeite block. The sample is stored specimen in NMNS (register number: NSM-R-133439), which is collected from around N36°04.6’/E139°11.9’). Zircons are fine-grained euhedral to subhedral crystals with length of 100 to 300 microns.

Analytical Methods and Results

Sample preparations

Zircon grains in the samples from KC, NS and YR were separated by standard crushing and heavy-liquid techniques and then handpicked to purify them. Zircon from IT was cut off from a thick polished section of the jadeitite sample. Two zircon grains of OS were picked up from a weathered jadeitite. The jadeitite chip including zircon (IT), half-cut large zircon grains (OS) and standard zircon (QGNG: 238U–206Pb* age of 1842.0±3.1 Ma; Black et al., 2003) were mounted in an epoxy resin and were polished. Fine-grained zircon grains (KC, NS and YR) and QGNG were mounted in another epoxy resin and were polished till the surface was flattened with the center of the embedded grains exposed for SEM and SHRIMP analyses.

Texture and inclusion observations in zircon

Both backscattered electron (BSE) images and cathodoluminescence (CL) images were used to select the sites for SHRIMP analysis. Qualitative analysis of mineral inclusions in zircon was con-
ducted by SEM-EDS, JEOL JSM5400 with Link QX2000 installed at NMNS.

Coarse-grained zircons from IT and OS show no clear compositional zoning on BSE images. However, the CL image of the zircon from IT sample shows a fragmented texture, and zircons from OS have a clear zoning pattern (Fig. 3a and b). No inclusion in the zircons has yet been observed in this study. On the other hand, fine-grained zircons in the samples KC, NS and YR show oscillatory zoning on BSE and/or CL images (Fig. 3c–e) which are commonly observed not only in igneous zircons (e.g. Corfu et al., 2003) but also hydrothermal zircons (Dubińska et al., 2004; Tsujimori et al., 2005). Zircons have mineral inclusions. Especially, zircon in the sample KC is remarkable. The zircons in sample KC have quartz–feldspar–mica aggregate (Fig. 2a and b) and/or apatite as an inclusion. The aggregates are thought to be crystalline melt inclusions (e.g. Thomas et al., 2003) probably originated from felsic melt based on their mineral assemblage. The NS zircons have inclusions consisting mainly of Si, Na ad Al with subordinate amounts of Mg, Fe and K (Fig. 2c). YR zircons have inclusions consisting mainly of Si and Al with subordinate amounts of K, Mg and Fe (Fig. 2d) but inclusions in NS and YR zircons are too small to identify their minerals.

**SHRIMP U-Pb dating of zircons**

U-Pb dating of zircons from the samples was carried out using SHRIMP II installed at Hiroshima University, Japan. The experimental conditions and the procedures follow Sano et al. (2000) and references therein. The spot size of the primary ion beam was approximately 20 µm. The 206 Pb/238 U ratios of the samples were calibrated using the empirical relationship described by Claoué-Long et al. (1995). The concentration of U in each analyzed spot was calibrated against an external standard SL13, which has a U content of 238 ppm (Claoué-Long et al., 1995). A correction for common Pb was made on the basis of the measured 206 Pb/208 Pb and 232 Th/238 U ratios and the two-stage model for common Pb compositions proposed by Stacey and Kramers (1975). Errors of all mean ages in this study are stated as

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**Fig. 2.** a) and b) Transmission light (TL) and backscattered electron (BSE) images of zircons including melt inclusions (MI), and BSE images of MI in sample KC (KC19 and a unanalyzed grain). Mineral assemblages of the MIs are estimated quartz (Qtz), K-feldspar (K-f), plagioclase (Pl) and mica. c) BSE image of NS02 with a Na-rich inclusion (Inc) consists of Si, Na, Al, Mg, Fe and K d) BSE image of YR01 with albite and Na-free inclusions (Inc) consist of Si, Al, K, Mg and Fe.
95% confidence interval.

Table 1 and 2 list zircon data in terms of U and Th concentrations, $^{204}\text{Pb}/^{206}\text{Pb}$, $^{207}\text{Pb}/^{206}\text{Pb}$, $^{208}\text{Pb}/^{206}\text{Pb}$, $^{238}\text{U}/^{206}\text{Pb}$, Th/U ratios, and $^{238}\text{U}/^{206}\text{Pb}^*$ ages. All errors are 1 sigma level. Sub-numbers of labels such as KT1.01 to KT1.10 in the tables indicate different pits in a single grain. Figure 5 and 6 show Tera–Wasserberg concordia diagrams and $^{238}\text{U}/^{206}\text{Pb}^*$ mean ages for all analyzed spot, calculated by Isoplot 3/Ex (Ludwig, 2003).

In a zircon from IT, age data were scattered ranging from 525 to 427 Ma; the weighted mean of these ages yields 497±23 Ma (Fig. 5a). Zircon age data for OS were scattered ranging from 540 to 487 Ma and two grains of OS1 (6 data) and OS2 (7 data) yield different mean ages; 496±12 Ma and 532±17 Ma, respectively (Fig. 5b and

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Fig. 3. Cathodoluminescence images (CL) of typical zircon grains. Ellipses on the images point to analyzed spots by SHRIMP with the sub-numbers.
5c). Most of their U and Th concentrations are remarkably low (Fig. 4). Zircon ages of KC were scattered ranging from 520 to 451 Ma and weighted mean of these ages is 501±5 Ma (Fig. 6a). NS and YR zircons yield younger mean ages, 136±5 Ma and 159±5 Ma, respectively (Fig. 6b and 6c).

**Discussion**

Multi-stage formation of zircons in jadeite rocks from Itoigawa (IT) and Osayama area (OS)

Zircons in jadeite rocks sometimes have inclusions indicating high-pressure condition (e.g. Tsujimori et al., 2005; Bröcker and Keasling, 2006), but it is not always for all jadeite rock (e.g. Shi et al., 2008). In this study, we couldn’t find inclusions in zircons from IT and OS. Kunugiza et al. (2002) concluded that zircons in jadeitite from Itoigawa area were formed coincidentally with jadeite because of their euhedral shape, presence in zeolite matrix which also contains euhedral jadeite and absence of negative Eu anomaly in zircon. Tsujimori et al. (2005) reported jadeite inclusion in zircon in jadeitite from Osayama area. According to these report, it is probable that the coarse-grained zircons in this study also formed coincidentally with jadeite. The age of each spot in the zircon from IT yields a mean age of 497±23 Ma, in agreement with previously reported values (519±17 Ma and 512.3±6.9 Ma; Kunugiza et al., 2002). But two zircons from sample OS (OS1 and OS2) yield different mean ages, 496±12 Ma and 532±17 Ma. Individual zircon spot ages of OS scattered from 540 to 487 Ma, and zircon age data from four zircon grains of Tsujimori et al. (2005) also scattered from 527 to 447 Ma. Although Tsujimori et al. (2005) concluded that the mean age of

### Table 1. SHRIMP U–Pb data and calculated ages of course zircons.

<table>
<thead>
<tr>
<th>Labels</th>
<th>(^{206}\text{Pb}/^{206}\text{Pb}</th>
<th>(^{207}\text{Pb}/^{206}\text{Pb}</th>
<th>(^{208}\text{Pb}/^{206}\text{Pb}</th>
<th>(^{208}\text{U}/^{206}\text{Pb}</th>
<th>\text{U (ppm)}</th>
<th>\text{Th (ppm)}</th>
<th>\text{Th/U}</th>
<th>(^{238}\text{U}/^{206}\text{Pb}^*</th>
<th>\text{age (Ma)}</th>
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<td>IT01</td>
<td>0.000697±0.000089</td>
<td>0.0626±0.0012</td>
<td>0.0422±0.0020</td>
<td>11.94±0.60</td>
<td>52</td>
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<td>0.11</td>
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<td>0.0737±0.0017</td>
<td>12.78±1.01</td>
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<td>IT03</td>
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<td>0.0585±0.0026</td>
<td>0.0203±0.0008</td>
<td>13.68±1.95</td>
<td>215</td>
<td>11</td>
<td>0.05</td>
<td>454.0±62.6</td>
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</tr>
<tr>
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<td>0.0761±0.0002</td>
<td>0.0838±0.0008</td>
<td>14.25±1.39</td>
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<td>3</td>
<td>0.11</td>
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<td>0.0363±0.0027</td>
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<td>114</td>
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<td>0.0668±0.0018</td>
<td>0.0513±0.0029</td>
<td>12.35±1.34</td>
<td>33</td>
<td>3</td>
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<td></td>
</tr>
<tr>
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<td>12.95±1.65</td>
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<td>0.12</td>
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<tr>
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<td>0.0740±0.0022</td>
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<td>43</td>
<td>8</td>
<td>0.20</td>
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<td>12</td>
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\(^{206}\text{Pb}^*\) and \(^{207}\text{Pb}^*\) mean radiometric \(^{206}\text{Pb}^*\) and \(^{207}\text{Pb}^*\), respectively. All errors are stated at 1 σ.
472 ± 8.5 Ma indicate age of jadeitization, we propose a possibility that jadeitization or zircon formation process in Osayama area is not a single event but multiple events from about 540 to 450 Ma. Actually, Fu et al. (2010) reported two types of zircons are recognized in jadeitite from Osayama area by observation of cathodoluminescence image and measurement of oxygen isotopes. On the other hand, three zircon ages are recognized in Myanmar jadeitite; Shi et al. (2005) interpreted the ages as inherited, jadeitite formation and thermal event.
Although it is difficult to conclude the origin of zircons in the Renge Belt, it is important that the approx. 500 Ma age has been recorded in the rocks from the Renge Belt.

**Jadeite rock from Kochi area (KC)**

The protolith of the KC sample is considered to be felsic igneous rock which age is 501 ± 5 Ma because some zircons have felsic melt inclusions (Fig. 2a and b) and zircon ages concentrate enough to interpret the mean age to igneous age of the protolith (Fig. 6a). Th/U ratios (Hoskin and Black, 2000) and inner structure of oscillatory zonings (Corfu et al., 2003) also support this idea. Formation of jadeite requires high-pressure condition (reviewed in e.g. Harlow and Sorensen, 2005) while felsic igneous rock was formed in island arc or continental crust. Therefore, the KC sample is interpreted as parentally a crustal material and once subducted (e.g. Clift and Vannucchi, 2004) to certain depth of jadeite formation. The K–Ar age of a jadeite-bearing schist from the Kurosegawa Belt is around 208–240 Ma (Maruyama et al., 1978), far younger than the zircon age of 501 ± 5 Ma. The former age will be corresponding to metamorphic stage, i.e. jadeitization, whereas the latter to the protolith igneous stage.

On the Japanese Islands, now, around 500 Ma igneous rocks are scarce; the Daiouin granitoid (Hitachi area, Ibaraki Pref.) and Hikawa granitoid (Higo Belt, Kumamoto Pref.) which ages are 490.8 ± 6.1 Ma; 502.5 ± 9.6 Ma, respectively (Sakashima et al., 2003). It is probable that these 500 Ma igneous rocks and associated rocks (Higo belt and Hitachi metamorphic rocks) were residues of missing crusts. Yokoyama et al. (2009) reported that detrital monazite age distribution of the Maizuru Belt (Permian to Triassic) have distinct peaks of 504–511 Ma. Although it is difficult to compare zircon age with monazite age, it is evident that 500 Ma crust existed extensively at least until Triassic in time. In spite of similar ages of zircons in the jadeite from the Renge Belt and jadeite-bearing rocks from the Kurosegawa Belt, their origins would be different each other.

**Mesozoic zircon ages of jadeite rocks in Nishisonogi (NS) and Yorii (YR) areas**

Zircons in NS and YR samples show oscillatory zoning on BSE and/or CL images (Fig. 3d and e) which are commonly observed not only in igneous zircons (Corfu et al., 2003) but also in hydrothermal zircons (e.g. Dubińska et al., 2005; Tsujimori et al., 2005). Hoskin and Black (2000) suggested that higher Th/U ratio (>0.1) of zircon is igneous in origin (e.g., Hoskin and Black, 2000). However, hydrothermal zircon also yield higher Th/U ratio (e.g. Tsujimori et al., 2005). Origins of zircons in the NS and YR samples are interpreted to be protolith or hydrothermal reaction. The zircons in the NS sample have mineral inclusions which compositions are relatively Na-rich, consisting of Si, Na, Al, Mg, Fe and K, whereas zircon inclusions in the YR sample consist of Si, Al, K, Mg and Fe. Although they are too small to identify the minerals, it is important whether the inclusions are Na-rich or not because it has been considered that there is strong relation between formation of jadeite rocks and Na–Al–Si rich fluid (Harlow and Sorensen, 2005; Tsujimori et al., 2005).

Associated metamorphic rocks have muscovite K–Ar ages of about 60–90 Ma for NS (Hattori and Shibata, 1982) and YR (Miyashita and Itaya, 2002; Hirajima et al., 1992). The zircons in the NS and YR are 136 ± 5 Ma and 159 ± 5 Ma, re-
respectively, far younger that the zircons as mentioned above. Metamorphic conditions of the jadeite rocks are within a stability field of jadeite–quartz assemblage, and are different from those of the associated metamorphic rocks in NS and YR. The latter conditions are within a stability field of albite. Although it is difficult to discuss the origin of zircons of NS and YR samples, it is important that the zircon ages of jadeite rocks of NS and YR are far younger than jadeite rocks of the Renge Belt and the Kurosegawa Belt.

Conclusions

The protolith of the KC sample is thought to be a ∼500 Ma felsic igneous rock and is interpreted as a continental material which once subducted to certain depth of jadeite formation. The zircon ages of IT are concentrated and yield a mean age of 497±23 Ma, in agreement with pre-
viously reported values. But two zircons from sample OS (OS1 and OS2) yield different mean ages, $496 \pm 12$ Ma and $532 \pm 17$ Ma, respectively. Although individual zircon spot ages of OS were scattered from 540 to 487 Ma as well as the previous report from 527 to 447 Ma, it is difficult whether they show igneous stage or metamorphic stage. Zircons from NS and YR are far younger, $136 \pm 5$ Ma (NS) and $159 \pm 5$ Ma (YR), than those from the Renge Belt and the Kurosegawa Belt. It is probable that these young ages show igneous ages because of existence of felsic melt inclusions and concentrated zircon ages.

All the ages obtained are not corresponding to the K–Ar ages of the associated metamorphic rocks. As the fine-grained zircons from three localities show apparently igneous zoning, it is probable that the ages are of the igneous stage different from the jadeitization stage of the parental rocks. Ages around 500 Ma have been
scarcely described from the Japanese Islands. Referring to the age data of the East Asia, the zircons with 500 Ma had belonged probably to the Jiamusi and Khanka blocks with ages around 500 Ma. The zircons with 136 Ma and 159 Ma from Kochi and Yorii, respectively, are enigmatic because of absence of the age in the Japanese Islands and Korean Peninsula. Further study will be necessary to discuss the origin of these zircons.

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