# U-Pb Dating of Large Zircons in Low-Temperature Jadeitite from the Osayama Serpentinite Mélange, Southwest Japan: Insights into the Timing of Serpentinization

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#### Abstract

Crystals of zircon up to 3 mm in length occur in jadeitite veins in the Osayama serpentinite mélange, Southwest Japan. The zircon porphyroblasts show pronounced zoning, and are characterized by both low Th/U ratios (0.2–0.8) and low Th and U abundances (Th = 1–81 ppm; U = 6–149 ppm). They contain inclusions of high-pressure minerals, including jadeite and rutile; such an occurrence indicates that the zircon crystallized during subduction-zone metamorphism. Phase equilibria and the existing fluid-inclusion data constrain P-T conditions to P > 1.2 GPa at T < 350°C for formation of the jadeitite. Most U/Pb ages obtained by SHRIMP-RG are concordant, with a weighted mean  $^{206}$ Pb/ $^{238}$ U age of 472 ± 8.5 Ma (MSWD = 2.7, *n* = 25). Because zircon porphyroblasts contain inclusions of high-pressure minerals, the SHRIMP U-Pb age represents the timing of jadeitite formation, i.e., the timing of interaction between alkaline fluid and ultramafic rocks in a subduction zone. Although this dating does not provide a direct time constraint for serpentinization, U-Pb ages of zircon in jadeitite associated with serpentinite result in new insights into the timing of fluid-rock interaction of ultramafic rocks at a subduction zone and the minimum age for serpentinization.

### Introduction

ZIRCON OCCURS as an accessory phase in igneous and metamorphic rocks and is the best mineral for U-Pb age dating, with a closure temperature around 900°C (Cherniak and Watson, 2001). On the other hand, in low-temperature (LT) environments such as blueschist- and greenschist-facies conditions, new zircon growth and overgrowth are very rare. Hence U-Pb dating of zircon in subduction-zone metamorphic rocks has been very limited. However, crystallization of zircon from hydrothermal fluid has been documented (e.g., Rubin et al., 1989; Rubatto and Hermann, 2003). In such LT environments, hydrothermal fluid may cause partial dissolution of preexisting zircon and precipitation of neoblastic zircon (e.g., Hansen and Friderichsen, 1989; Sinha et al., 1992; Vavra et al., 1999). These processes accompany Pb or U loss and cause isotopic discordance at LT conditions.

Serpentinite is a unique environment for precipitation of hydrothermal zircon. Zircon has been reported from serpentinite-related LT metasomatic rocks such as rodingite blackwall and jadeitite (Kobayashi et al., 1987; Harlow, 1994; Bröcker and Enders, 1999; Miyajima et al. 2001; Dubińska et al. 2004); reconnaissance U-Pb dating for such LT zircon has just began (e.g., Bröcker and Enders, 1999; Kunugiza et al., 2002; Dubinska et al., 2004). Isotopic and chemical information on these zircon crystals provide a key not only to understand the fluid history concerning serpentinization but also to evaluate the hydrothermal effects on both growth and dissolution of zircon at LT conditions.

In this contribution, we present SHRIMP (sensitive, high-resolution ion microprobe) U-Pb isotopic data for large hydrothermal zircon crystals, up to 3 mm in size, in jadeitite (nearly monomineralic

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FIG. 1. Simplified map of the Chugoku Mountains of Southwest Japan, showing various pre-Triassic geotectonic units and the sample locality.

jadeite-rich rock) from a serpentinite-matrix mélange in the Osayama Mountain, Southwest Japan. Although zircon porphyroblasts from this locality have been previously considered as a relict igneous phase (Miyamoto and Yanagi, 1998), we have recently found a few jadeite inclusions in zircon. We also describe the internal texture and U-Th chemistry of hydrothermal zircon, discuss the implications for the zircon crystallization in jadeitite, and comment on the U-Pb age dating for serpentinization at a subduction zone.

#### **Geologic Outline**

Jadeitite, omphacitite, albitite, rodingite, and blueschist-facies schist have been identified as blocks in the Osayama serpentinite mélange of the central Chugoku Mountains (Tsujimori, 1997; Tsujimori and Itaya, 1999; Tsujimori and Liou, 2004a, 2005a, 2005b) (Fig. 1). Serpentinized peridotite bodies and related mélanges occur as an Early Paleozoic ophiolitic nappe that occupies the highest structural position of the Oeyama belt (Ishiwatari and Tsujimori, 2003). Chrysotile and lizardite are major serpentine-group minerals in the Osayama mélange (Tsujimori, 1998). Protoliths of the serpentinites consist mainly of harzburgite with minor dunite, podiform chromitite, and gabbroic dikes (e.g., Arai, 1980); the gabbroic intrusives with MORB-like geochemical signatures yield Sm-Nd isochron ages of ca. ~560 Ma (Hayasaka et al., 1995). Phengites from blueschistfacies schists at Osayama yield K-Ar ages of 327–273 Ma (Tsujimori and Itaya, 1999).

Jadeitites in the Osayama serpentinite mélange occur as veins up to 4 m wide. They are composed mainly of jadeite (generally 75% by volume) with minor amounts of grossular (nearly pure), and trace rutile and zircon. The jadeite-rich matrix is heterogeneous and shows variable degrees of retrogression. Omphacite, analcime, pectolite, stronalsite, thomsonite, natrolite, vesuvianite, prehnite, phlogopite, serpentinite, and titanite are retrograde phases (Kobayashi et al., 1987; Tsujimori, 1998). Shoji and Kobayashi (1988) described primary fluid inclusions in jadeite. Preliminary U-Pb ages of large zircon porphyroblasts in jadeitite using convectional thermal ionization mass spectrometry yield ca. 500-450 Ma (Miyamoto and Yanagi, 1998); the data was interpreted as the crystallization age of an igneous protolith for the jadeitite.

# Sample Description and Condition of Zircon Crystallization

Electron microprobe analysis for rock-forming minerals was carried out with a JEOL JXA-8900R at



FIG. 2. Microtexture of the investigated jadeitite. A. Plane polarizer view of the occurrence of jadeite (Jd) with grossular (Grs). B. Plane polarizer view of occurrences of zircon (Zrn); matrix minerals including jadeite (Jd) and secondary pectolite (Pct) are also shown. C. Cathodoluminescence image of zircon of (B). Texture shows zircon consisting of at least two crystals.

Okayama University of Science. Quantitative analyses were performed with 15 kV accelerating voltage, 12 nA beam current, and a 3 µm beam size. The ZAF (oxide basis) method was employed for matrix corrections. Cathodoluminescence (CL) imaging was obtained using a JEOL 5600LV Scanning Electron Microscope (SEM) equipped with a HAMAMATSU photo multiplier tube at Stanford University.

In the investigated sample, jadeite with 91-99 mol% NaAl<sub>2</sub>Si<sub>2</sub>O<sub>6</sub> (Jd) component occurs as either subhedral to anhedral prismatic crystals or radial aggregates (Figs. 2A and 3); most are less than 0.5 mm in length, but some are up to 5 mm. Jadeite

contains rare grossular, zircon, and rutile as tiny inclusions (< 0.1 mm). Some secondary omphacites (39–58 mol% Jd) epitaxially overgrow jadeite crystals and fill fractures together with analcime (Fig. 3). In the altered sections of the sample, jadeite was extensively replaced by pectolite. Coarse-grained rutile prisms, up to 4 mm long, occur in the jadeitedominant matrix and are rimed by secondary titanite that is in contact with secondary pectolite.

Zircon occurs as discrete euhedral to subhedral crystals up to 3 mm in length or as twinned crystals or aggregates in the matrix (Fig. 2B and 2C); zircon contains tiny inclusions of rutile and rare jadeite (97 mol% Jd; Fig. 3). Fine-grained zircon also occurs as



FIG. 3. Compositions of sodic pyroxenes from the investigated jadeitite on the jadeite (Jd)–acmite (Ac)–diopside + hedenburgite (Di + Hd) ternary diagram. The  $Fe^{2+}/Fe^{3+}$  ratio for clinopyroxene was estimated assuming a cation total of 4.00.

inclusions in a single jadeite crystal (Kobayashi et al., 1987). This evidence indicates synchronous growth of zircon and jadeite.

Jadeitite formation requires HP condition (P > 0.5–0.6 GPa at T =  $200-400^{\circ}$ C) and infiltration of Na- and Al-rich alkaline fluid (Harlow, 1994; Shi et al., 2003). It is thought that zircon crystals directly crystallized from a hydrothermal fluid during the jadeitite formation in serpentinites. In the Osavama jadeitite, the homogenization T of fluid inclusions in jadeite examined by Shoji and Kobayashi (1988) requires a minimum of ~345°C for crystallization of the primary jadeitite minerals. Thermodynamic calculation employing the THERMOCALC program (ver. 3.21) (Powell et al., 1998) indicates P > 1.2 GPa at a nominal  $T = \sim 350^{\circ}$ C for the primary jadeite + rutile + grossular assemblage, and P = 0.4-0.6GPa at 200-300°C for the secondary omphacite + analcime assemblage. The presence of grossular and absence of carbonate minerals suggest low X<sub>CO2</sub> of the hydrothermal fluid.

# U-Th Chemistry and U-Pb Isotopic Results

U-Th-Pb analyses were performed with the SHRIMP-RG (sensitive high-resolution ion microprobe-reverse geometry) in the Stanford-USGS cooperative ion microprobe facility at Stanford University. Instrumental conditions and data acquisition are similar to the procedures described by Williams (1998). Analytical spots ~ 40 µm in diameter were sputtered using an ~5 nA  $O_2^-$  primary beam. The data were collected in sets of six scans through nine mass spectra. The primary beam was rastered across the analytical spot for 120 s before

analysis to reduce surficial common Pb resulting from sample preparation and Au-coating. Concentration data were calibrated against CZ3 zircon (550 ppm U), and isotope ratios were calibrated against R33 (419 Ma, John Aleinikoff, pers. commun., 2002). Data reduction follows Williams (1998), and utilized Squid (Ludwig, 2001). Isoplot 3 (Ludwig, 2003) was used to calculate all ages, which are reported here at the 95% confidence level.

Representative CL images of three zircon grains are shown in Figure 3; the Th and U abundance from 28 spots of 8 zircon grains are presented in Figure 4. The dating results are shown in Table 1. As shown in the images of Figure 4, zircon porphyroblasts in the Osayama jadeitite have preserved remarkable internal zoning textures including fine, euhedral growth layers with sharp contrasts in CL brightness; these zircons are characterized by low Th and U abundances. Based on textural and geochemical features, analyzed zircons are subdivided into two different zones. Type I is a normal growth zone that is commonly characterized by a broad, stubby core with concentric oscillatory-zoned rims. This texture, shown in Figure 4A, is similar to the growth pattern of hydrothermal zircon crystallized from fluid in serpentinite (e.g., Fig. 7 of Dubińska et al., 2004), and provides evidence of crystallization from fluid. Type I zones in zircon crystals show a wide geochemical variation; Th and U exhibit a positive correlation—Th increases from 3 to 37 ppm as U increases from 11 to 82 ppm. However, concentrations fluctuate greatly from one layer to another (Figs. 4A and 5A). Type II zones in zircon crystals, with dark CL, represent texturally inherited cores rarely preserved in Type I zones (Fig. 4B) and have



FIG. 4. Cathodoluminescence images of representative zircon separates from the Osayama jadeitite showing growth zoning layers and three zircon zones described in the text. Ellipses within crystal represent primary beam pits; SHRIMP U-Pb results are also shown as apparent  $^{206}Pb/^{238}U$  ages (Ma) with  $1\sigma$  errors. For explanation see text. Scale bar represents 200 µm.

very irregular zoning patterns; Th/U ratios of Type II zones are significantly higher (0.70–0.80) than that of the others (0.21–0.48) (Fig. 5B). Textures and different Th/U ratios indicate partial dissolution of early-formed zircon crystals.

The analyzed U-Pb isotopic ratios are plotted on a Tera-Wasserburg diagram with  $1\sigma$  errors (Fig. 6). Most of the data points are concordant within error. Twenty-five analyses of the Type I zone yields <sup>206</sup>Pb/ <sup>238</sup>U ages arranging 452 to 521 Ma, whereas three analyses of the Type II zone yields 488 to 523 Ma (Table 1). Although the <sup>206</sup>Pb/<sup>238</sup>U ages are slightly scattered within the same zones in same zircon crystals (Fig. 4), the lack of noticeable correlation between apparent <sup>206</sup>Pb/<sup>238</sup>U age and either U abundance or Th/U ratio (Fig. 5) indicates that the observed chemical variations may not be related to zircon alteration and the U-Pb system may not be significantly disturbed by later events. When the Type II zone is excluded, the type I zone yields a weighted mean <sup>206</sup>Pb/<sup>238</sup>U age of 472 ± 8.5 Ma (MSWD = 2.7), which is in agreement with the conventional data (Fig. 6).

# Zircon Growth in Jadeitite from Serpentinite

Most ultramafic rocks contain less than 20 ppm Zr (Erlank et al., 1978); this value is less than 2% of the Zr content of highly evolved granite (>1000 ppm). In fact, zircon in ultramafic rocks is itself very rare except for a few metasomatized peridotites from the Kokchetav Massif, Kazakhstan (e.g., Katayama et al., 2003). Despite such Zr-depleted environments, however, many zircon occurrences in jadeitite and rodingite in serpentinized mantle peridotites have been commonly documented, as mentioned above. On the other hand, formation of nearly monomineralic jadeitite is rare but is almost always restricted to serpentinites from blueschist or eclogite terranes, clearly indicating a subductionzone environment with a low geotherm (e.g., Coleman, 1961; Harlow, 1994). It is well known that the hydration of ultramafic rocks results in high-pH alkaline fluid (pH ~10) in serpentinites from both orogenic belts and modern seafloor environments (e.g., Coleman, 1971; Kelley et al., 2001). The fluidinclusion studies of Guatemalan jadeitites (Johnson

											<sup>207</sup> Pb/ 206DL		
		U	Th	P <sup>brad</sup> *	*f		<sup>238</sup> U/		<sup>207</sup> Pb/		age**		
Label	Zone	(ppm)	(ppm)	(ppm)	(%)	Th/U	<sup>206</sup> Pb	Error	<sup>206</sup> Pb	Error	(Ma)	Error	
Grain 01													
#01-1	Type I	11.1	27	0.8	2 23	0.25	12.00	42	0.0686	10.1	509.0	21.3	
#01-2	Type I	11.7	2.4	0.7	-1.48	0.21	13.62	4.0	0.0609	10.8	454.2	18.2	
Grain 05													
#05-11	Type I	15.4	37	11	-0.96	0.25	12 21	34	0.0660	85	502.2	16.9	
#05-13	Type I	12.5	3.4	0.8	4.79	0.28	13.56	4.1	0.0668	12.4	452.8	18.8	
Grain 07													
#07-34	Type I	21.8	64	14	-0.78	0.30	13 13	31	0.0603	81	471.1	14.3	
#07-35	Type I Type I	18.2	4.9	1.3	-0.76	0.28	12.37	3.1	0.0614	9.6	498.7	15.8	
#09-15 (34-3)	Type I	60.4	20.5	3.0	1.96	9 (F1g. 5) 0 35	A) 13 31	<b>?</b> ?	0.0600	4.0	460.0	10.3	
#09-17 (3A-7)	Type I	46.3	14.2	3.2	3.15	0.32	12.26	2.6	0.0020	82	494.7	13.0	
#09-18 (3A-6)	Type I	78.8	26.6	4.9	0.10	0.35	13.87	2.0	0.0601	4.6	446.5	9.3	
#09-19 (3A-5)	Type I	53.2	17.4	3.4	2.04	0.34	13.64	2.3	0.0601	5.6	454.0	10.6	
#09-20 (3A-4)	Type I	58.5	19.9	3.7	1.12	0.35	13.58	2.2	0.0602	5.1	455.7	10.1	
#09-21 (3A-1)	Type I	94.1	37.3	6.0	0.89	0.41	13.58	2.0	0.0600	4.1	455.9	9.0	
#09-22 (3A-2)	Type I	58.8	20.1	3.8	-0.34	0.35	13.48	2.3	0.0583	5.4	460.2	10.4	
#09-23 (3A-8)	Type I	81.8	29.5	5.3	0.69	0.37	13.36	2.0	0.0593	4.1	463.6	9.2	
Grain 10													
#10-24	Type I	13.1	3.5	0.9	4.41	0.27	12.72	3.97	0.0682	10.7	481.1	19.2	
#10-27	Type I	11.0	2.9	0.8	-1.22	0.27	12.21	3.69	0.0692	9.3	500.0	18.5	
Grain 11 (Fig. 3B)													
#11-3	Type II	85.2	68.0	6.3	-0.149	0.82	11.70	1.89	0.0605	3.5	526.9	9.8	
#11-4	Type I	17.7	4.7	1.2	-0.804	0.27	12.18	3.14	0.0550	8.5	510.3	15.9	
#11-5	Type I	17.3	3.6	1.2	-0.897	0.21	12.52	3.20	0.0659	8.5	490.0	15.7	
#11-6	Type I	25.4	7.5	1.6	-0.586	0.30	13.39	2.86	0.0611	6.8	461.5	13.1	
#11-7	Type II	63.5	45.0	4.3	-0.256	0.73	12.69	2.12	0.0588	4.7	487.7	10.2	
#11-36	Type I	26.7	7.9	1.9	-0.536	0.30	11.77	2.73	0.0651	6.5	521.0	14.2	
Grain 14													
#14-28	Type I	10.4	2.2	0.7	4.56	0.22	12.669	3.94	0.0575	10.3	489.4	19.2	
#14-30	Type I	60.1	24.9	4.0	-0.28	0.43	12.994	2.16	0.0572	4.9	477.6	10.2	
#14-31	Type I	46.4	21.7	3.1	-0.37	0.48	12.853	2.34	0.0633	5.5	479.2	11.2	
#14-32	Type I	23.1	6.5	1.5	-0.70	0.29	13.217	2.93	0.0541	8.0	471.5	13.7	
	Grain 15												
#15-37	Type I	26.1	7.0	1.8	-0.52	0.28	12.204	2.70	0.0613	6.5	505.3	13.6	
#15-38	Type II	83.7	61.8	5.9	0.39	0.76	12.210	1.93	0.0596	3.8	506.1	9.6	

# TABLE 1. Ion-Probe U-Th-Pb Isotope Data of Zircon in Jadeitite from the Osayama Serpentinite Mélange, Southwest Japan1

<sup>1</sup>All errors are 1  $\sigma$  of standard deviation. \* = percentage of common <sup>206</sup>Pb in total measured <sup>206</sup>Pb. A negative value indicates that the number of ion counts was indistinguishable from background. In such case no common Pb correction is made; \*\* = common Pb correction based on <sup>207</sup>Pb.



FIG. 5. Plots of apparent <sup>206</sup>Pb/<sup>238</sup>U ages (Ma) and Th and U abundance (ppm). Labeled data within plot 5A relate to analyses of zircon shown in Figure 3A. For explanation see text.

and Harlow, 1999) revealed that the infiltration of seawater-like, high-salinity fluid (up to 8 wt% NaCl equivalent) into serpentinites might have played a significant role in jadeitite formation. It is thus possible that serpentinization and subsequent migration of high-pH, high-salinity fluid in serpentinites were almost contemporaneous. Moreover, some geologic and experimental evidence for Zr mobility in hydrothermal environments indicates that Si-undersaturated fluid containing F, Cl, Na, and Ca can behave as a corrosive solution that promotes high concentrations of high-field-strength elements (HFSEs) (e.g., Hansen and Friderichsen, 1989; Sinha et al., 1992; Vard and Williams-Jones, 1993; Rubatto and Hermann, 2003).

Considering these synthetically, fluid-rock interaction associated with the serpentinization of ultramafic rocks appears to be the only reasonable process that may account for the Zr concentration and for the precipitation of zircon. Although the source of Zr remains unclear, Zr saturation was most likely supplied by infiltration of an HFSE-enriched subduction-zone fluid or internal source such as clinopyroxene breakdown during serpentinization, inasmuch as mantle peridotite clinopyroxene contains 10–30 ppm Zr (e.g., Batanova and Sobolev, 2000). In any case, such hydrothermal fluid may have contributed to zircon crystallization during serpentinization in the subduction-zone environment.

# Time Constraints on Fluid-Rock Interaction

The investigated zircon porphyroblasts have been previously considered as a relict igneous phase (Miyamoto and Yanagi, 1998). Our work has confirmed the result of earlier study that a zircon U-Pb age dated by conventional technique is Ordovician. However, we have also revealed syn-metamorphic, simultaneous growth of zircon with jadeite from the serpentinite-related hydrothermal fluid at a condition of P > 1.2 GPa at T < 350°C. Inasmuch as the P-T condition of zircon precipitation is well below the closure temperature of the zircon U-Pb system, we interpret the SHRIMP U-Pb ages as the timing of zircon crystallization from alkaline fluid during the formation of the jadeitite. As mentioned above, the fluid circulation in ultramafic rocks and the subsequent fluid-rock interaction under the LT-HP condition are required to form nearly monomineralic



FIG. 6. Tera-Wasserburg diagram for SHRIMP analyses of zircons from the Osayama jadeitite. The grey ellipses represent conventional data by Miyamoto and Yanagi (1998).

jadeitite. Thus, an age of 472 Ma may represent the timing of interaction between hydrothermal fluid and ultramafic rocks in a subduction zone. Fluid circulation may have resulted in varying degrees of metasomatism leading to mineralization of jadeite and zircon during serpentinization. Despite its geologic or tectonic importance, however, radiometric dating of serpentinization is a difficult problem. Although this dating does not provide a direct time constraint for the serpentinization, U-Pb ages of zircon in jadeitite can result in new insights into the minimum age of serpentinization by the fluidrock interaction of ultramafic rocks at a subduction zone.

The hydrothermal mineralization of 472 Ma is significantly older than the K-Ar ages, 327–273 Ma, of blueschist-facies blocks from the Osayama serpentinite mélange. This suggests the jadeitite formation is not related to regional Late Paleozoic blueschist-facies metamorphism. In the eastern Chugoku Mountains, Early Paleozoic HP epidoteamphibolites with hornblende K-Ar age of 443–403 Ma occur in a serpentinized peridotite body located about 180 km to the east of the Osayama serpentinite mélange (Tsujimori and Liou, 2004b). The jadeitite formation may be related to this Early Paleozoic HP metamorphism.

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