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Zircon U–Pb dating, geochemistry and Sr–Nd–Pb–Hf isotopes of the Wajilitag alkali mafic dikes, and associated diorite and syenitic rocks: Implications for magmatic evolution of the Tarim large igneous province

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The Early Permian Tarim large igneous province (Tarim LIP) consists mainly of basaltic lavas, mafic–ultramafic intrusions including dikes and, syenite bodies in the Tarim Basin, NW China. A major unit of the Tarim LIP, the Wajilitag intrusive complex, consists of olivine pyroxenite, clinopyroxenite and gabbro units (from bottom to top), diorite and syenite rocks occurred in the upper part of the complex and alkali mafic dikes intrude the clinopyroxenite phase. Here we report the zircon U–Pb age and Hf isotopes, geochemical characteristics and Sr– Nd–Pb isotopic data of the alkali mafic dikes, and diorite, aegirine–nepheline syenite and syenite porphyry units in the Wajilitag intrusive complex. Zircons from the diorite and alkali mafic rocks yield concordant crystallization ages of 275.2 \pm 1.2 Ma and 281.4 \pm 1.7 Ma, respectively. The diorite and syenitic rocks in Wajilitag area have a narrow range of SiO₂ contents (51.9–57.3 wt.%), and are enriched in total alkalis (Na₂O + K₂O = 8.3–14.3 wt.%), among which the aegirine–nepheline syenite and syenite porphyry have the geochemical affinity of A-type granites. The alkali mafic rocks and syenitic rocks have high Al_2O_3 (19.4–21.1 wt.%), Zr, Hf, Ba contents, total rare earth element abundances and LREE/HREE ratios and low Mg# value, K, P and Ti contents. Diorites have lower Al2O3 contents, total REE abundances and LREE/HREE ratios and higher Mg# values than the alkali mafic rocks and syenitic rocks. The diorites and syenitic rocks have low initial 87 Sr/ 86 Sr ratios (0.7034–0.7046), and high ε Nd(t) values (0.1–4.1) and zircon ε Hf(t) values (−0.9–4.4). All the diorites and syenitic rocks show the ²⁰⁶Pb/²⁰⁴Pb ratios ranging of 18.0–19.5, ²⁰⁷Pb/²⁰⁴Pb of 15.4–15.6 and ²⁰⁸Pb/²⁰⁴Pb of 38.0–39.9. tios indicate a FOZO-like mantle source for the diorite and syenitic rocks, similar to that of the mafic–ultramafic rocks in the Wajilitag complex. In contrast, zircon Hf isotopes of basalt and syenite elsewhere in the Tarim LIP indicate a FOZO-like component may also contribute to Tarim LIP magmatism. Geochemical and Sr–Nd–Pb–Hf isotopic features reflect that diorites and syenitic rocks are probably derived from a FOZO-like mantle source, consistent with a plume mantle origin and then underwent crystal fractionation process.

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1. Introduction

The Tarim large igneous province (Tarim LIP), formed during the early Permian, is located in the Tarim Basin, the largest basin in China [\(Li et al., 2011; Z.L. Li et al., 2012; Yang et al., 2006, 2007; Yu, 2009;](#page-13-0) [Zhang et al., 2008\)](#page-13-0), and is mainly composed of basalts and numerous intrusions. It is comparable to the Siberian Traps and Emeishan large igneous province occurred in the Eurasian continent during the Permian.

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The Tarim basalts typically formed earlier than the Tarim intrusive rocks in a within-plate setting ([X. Yu et al., 2011; Zhou et al., 2009\)](#page-14-0).

The mid-ocean ridge basalts (MORB) and ocean island basalts (OIB) are considered as the result of mixing between several end-members, such as DMM (depleted MORB mantle), HIMU (high $\mu =$ high U/Pb), EMI (enriched mantle with low ε Nd(t) and ²⁰⁶Pb/²⁰⁴Pb, and intermediate initial 87 Sr/ 86 Sr), EMII (enriched mantle with intermediate εNd(t), and high $^{206}Pb/^{204}Pb$ and initial $^{87}Sr/^{86}Sr$) and FOZO (Focus Zone) [\(Hart, 1988; Hart et al., 1992; Hofmann, 2003; Saal et al., 2005;](#page-13-0) [Stracke et al., 2005; Tackley, 2007; Willbold and Stracke, 2010; Zindler](#page-13-0) [and Hart, 1986\)](#page-13-0). Previous studies revealed Keping basalts are derived from an OIB-like (EMII) components whereas the intrusive rocks originated from a mixing between the DDM and EMII end-member

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interpreted to represent the interaction between OIB-like asthenospheric (plume) mantle and subcontinental lithospheric mantle, based on their whole-rock geochemistry, Sr–Nd–Pb–Hf isotopes and PGE values [\(Li et al., 2011; Y.Q. Li et al., 2010, 2012; Z.L. Li et al., 2012;](#page-13-0) [Yang et al., 2007; Zhang et al., 2008; C.L. Zhang et al., 2010](#page-13-0)). While geochemistry and Sr–Nd isotopes for the mafic to ultramafic igneous rocks in the Xiaohaizi and Wajilitag areas of Bachu County have been studied well, the newly discovered alkali mafic dikes (mainly refers to alkali diabase) and diorite as well as syenitic and syenitic porphyry units of the Wajilitag complex should be further studied.

Alkali mafic dike, diorite and syenitic rocks have been identified as important units of some large intrusions in some LIPs [\(Shellnutt](#page-13-0) [et al., 2011; Song et al., 2008; Xu et al., 2008; Zhou et al., 2006\)](#page-13-0). The various diabase and bimodal dikes intruded into the strata and intrusions and always had great significance to restrict the relationship among different rock types ([Li et al., 2011; Shellnutt et al., 2012;](#page-13-0) [Yang et al., 2007\)](#page-13-0). For instance, diorite can often appear with gabbro in the intrusions and makes a vital contribution to ore deposit mineralization.

The Wajilitag complex (also known as the Bachu intrusions; [Zhang](#page-14-0) [et al., 2008](#page-14-0)) is a key intrusion in Tarim LIP. This paper presents new geochemistry, Sr–Nd–Pb–Hf isotopic data and zircon U–Pb dating of the new discovered diorite, nepheline-bearing syenite and syenite porphyry units in the upper part of the Wajilitag complex and the alkali mafic dikes cutting these units. These data will be integrated with published data for the mafic–ultramafic portions of the Wajilitag complex to provide a more complete interpretation of the magmatic source(s) and evolutionary history of this important Wajilitag complex of the Tarim LIP and with implications for the Tarim LIP as a whole.

2. Geological background

2.1. Regional geology

The Tarim Basin, surrounded by the Tianshan, Kunlun and Altun Mountains in the northwestern China [\(Fig. 1](#page-2-0)a, b), is underlain by a Precambrian crystalline basement and consists of Phanerozoic sedimentary strata ranging in age from Ordovician to Neogene [\(Chen](#page-13-0) [et al., 1997a,b; Jia, 1997; Jia et al., 1995; Jiang et al., 2004; Z.L. Li](#page-13-0) [et al., 2012; Yang et al., 2006, 2007; X. Yu et al., 2011; Zhang et al.,](#page-13-0) [2003; C.L. Zhang et al., 2013; D.Y. Zhang et al., 2013; Zhou et al.,](#page-13-0) [2009\)](#page-13-0). The Permian igneous rocks of the Tarim Basin have been well studied through a number of field geological investigations, drill core records, geophysical investigation and geochemical and geochronological studies ([Borisenko et al., 2006; Cao et al., 2014;](#page-13-0) [Chen et al., 1997a,b, 2010; Jia, 1997; Jia et al., 1995; Jiang et al.,](#page-13-0) [2004; Li, 2013; Y.Q. Li et al., 2010; Li et al., 2011; Y.Q. Li et al., 2012;](#page-13-0) [Z.L. Li et al., 2012; Tian et al., 2010; Wei et al., 2014; Xu et al., 2014;](#page-13-0) [Yang et al., 2006, 2007; Yu et al., 2009; J.C. Yu et al., 2011; X. Yu](#page-13-0) [et al., 2011; Yu et al., 2012; Zhang et al., 2003, 2008; C.L. Zhang](#page-13-0) [et al., 2010; D.Y. Zhang et al., 2010; Y.T. Zhang et al., 2010; D.Y.](#page-13-0) [Zhang et al., 2013; Zhou et al., 2009; Zou et al., 2013\)](#page-13-0). The Permian strata in the Tarim Basin are composed of volcanic sedimentary and sedimentary sequences, including limestones, sandstones, siltstones and basaltic lava. As the main phase of the Tarim LIP, the continental flood basalts are distributed over an area of more than $250,000$ km² [\(Yang et al., 2007](#page-14-0)). In addition, the mafic–ultramafic intrusions, kimberlitic intrusions, syenitic bodies, mafic and bimodal dikes discovered in Bachu area are also the parts of the Tarim LIP ([Cao et al.,](#page-13-0) [2014; Li et al., 2011; Y.Q. Li et al., 2012; Wei and Xu, 2011; Yang](#page-13-0) [et al., 2006, 2007; Yu et al., 2009; Zhang et al., 2008; D.Y. Zhang](#page-13-0) [et al., 2013; Zhou et al., 2009\)](#page-13-0). Some researchers suggested that the early Permian igneous event was probably caused by a Permian mantle plume upwelling under the Tarim Block ([Li, 2013; Li et al., 2008;](#page-13-0) [Z.L. Li et al., 2012; Zhang et al., 2008](#page-13-0)).

2.2. The Wajilitag complex

The intrusive complex is located in Wajilitag area of the southeastern Bachu County in the western Tarim Basin ([Fig. 1](#page-2-0)b), and has a layered structure with an outcropping area of ca. 15 km^2 ([Fig. 1](#page-2-0)c). It has been previously referred to as the Bachu layered intrusive complex ([Zhang et al., 2008](#page-14-0)), but herein is referred to as the Wajilitag ntrusive complex. The complex consists of a sill-like mafic–ultramafic layered intrusion, syenitic bodies, mafic dike swarms and bimodal dikes, and intruded into the Silurian–Devonian sedimentary rocks with contact zones dipping 20–40° toward the interior of the complex. The complex has a thickness of ca. 100–300 m and extends over an area of ca. 15 km^2 [\(Cao et al., 2014; Y.Q. Li et al.,](#page-13-0) [2012\)](#page-13-0). The intrusion consists of a sequence of olivine pyroxenite, clinopyroxenite and gabbro from bottom to top in the intrusion [\(Cao et al., 2014; Zhang et al., 2008](#page-13-0)). The Fe–Ti oxide deposit is mainly located in the clinopyroxenite unit, and contains 100 million tons of ore reserves with ca. 20 wt.% FeO_T, 7 wt.% TiO₂ and 0.14 wt.% V_2O_5 [\(Gao, 2007\)](#page-13-0). Apart from the layered intrusion, there are syenitic and diorite bodies outcropping above the mafic–ultramafic intrusion with a sharp contact. Numerous mafic and bimodal dikes intruded into the mafic–ultramafic layered intrusion and the host upper Devonian sedimentary rocks; these dikes have variable trends ([C.L.](#page-14-0) [Zhang et al., 2010\)](#page-14-0). A similar situation applies to the mafic dikes and bimodal dikes that crop out around the neighboring Xiaohaizi intrusive complex [\(Yang et al., 2007](#page-14-0)). Moreover, the Wajilitag complex also has some transitional rock types (e.g., plagioclase-bearing pyroxenite, olivine gabbro, hornblende-bearing syenite, aegirine– nepheline syenite, quartz syenite). On the basis of the geochemical and geochronological data of the intrusive rocks, [Zhang et al. \(2008\)](#page-14-0) suggested that the emplacement age of the Wajilitag (Bachu) complex was ca. 275 Ma and that it was a product of a Permian mantle plume emplaced at the base of the lithosphere below the Tarim Basin. [Yang](#page-14-0) [et al. \(2007\)](#page-14-0) proposed that the syenitic porphyry in the Xiaohaizi area formed in a within-plate environment at ca. 277 Ma and was derived from differentiation of mantle derived melts; and [Yu et al. \(2009\)](#page-14-0) reported the formation of mafic–ultramafic igneous rocks in the same area being genetically related to a mantle plume rising under Tarim Basin. Building on the previous studies on the mafic–ultramafic layered intrusion, these additional mafic dikes and quartz syenite all belonging to the Wajilitag intrusive complex, we analyzed the diorite and aegirine–nepheline syenitic bodies, alkali mafic dikes and bimodal dikes which intruded into the clinopyroxenite and syenite porphyry [\(Fig. 2a](#page-3-0), b, c).

3. Petrography of the Wajilitag alkali mafic diked rocks, diorite and syenitic rocks

The rocks of this study are distributed in the Wajilitag area ([Fig. 1](#page-2-0)c) and associated with the mafic–ultramafic rocks of the Wajilitag complex. The newly characterized units of this study include alkali diabase, syenitic porphyry, aegirine–nepheline syenite and diorite.

The aegirine–nepheline syenite consists of orthoclase (60–70%), nepheline (5–10%), aegirine (20%) and biotite (1–5%) as well as apatite, zircon and magnetite [\(Fig. 2](#page-3-0)d, e).

The diorite is mainly composed of plagioclase (60–70%), hornblende (10–15%), brown biotite (8–10%), augite (5–10%), opaque minerals (5–10%) and perthite (5%) with accessory zircon and apatite. Plagioclase exhibits characteristic zoning and twinning, and some plagioclase grains are surrounded by perthite. In addition, some hornblende grains are replaced by biotite ([Fig. 2](#page-3-0)f).

The alkali mafic dike has a microporphyritic texture ([Fig. 2g](#page-3-0)) with phenocrysts of plagioclase (30%) which display carlsbad–albite compound twining and compositional zoning. In the matrix, aegirine– augite (30–40%) and plagioclase (30–40%) exhibit a diabasic texture [\(Fig. 2h](#page-3-0)).

Fig. 1. Geotectonic units of (a) China (referred to Li. [2013](#page-13-0)) and (b) Tarim Basin (referred to Li et al., [2008](#page-13-0)) and (c) lithological sketch map of the Wajilitag area (the syenite porphyry, aegirine-nepheline syenite, diori located in the areas of I, II, III and IV, respectively).

Fig. 2. Field photos and microphotos of the samples in Wajilitag area: (a) aegirine-nepheline syenite bodies intruding into the Silurian-Devonian sedimentary rocks; (b) bimodal diabase and syenite porphyry dikes; (c) alkali mafic dike intruded into the clinopyroxenite; (d) and (e) petrography of aegirine-nepheline syenite with minerals of aegirine, nepheline and orthoclase; (f) zoned and subhedral structure of plagioclase in diorite; (g) and (h) large plagioclase phenocryst and aegirine–augite in alkali diabase, and (i) microcrystalline texture in syenite porphyry. Mineral abbreviations in the microphotos: aegirine (Aeg), nepheline (Ne), orthoclase (Or), hornblende (Hbl), biotite (Bt), plagioclase (Pl), and aegirine-augite (Agt).

The syenite porphyry has a microcrystalline texture typically with phenocrysts of orthoclase (20–30%). Orthoclase phenocrysts display corroded rims (Fig. 2i).

4. Analytical methods

4.1. Zircon U–Pb dating and Hf isotope analyses

The samples of the diorite (WJL050710) and alkali mafic rock (WJL081907) were selected for zircon U–Pb dating. Zircon grains were first separated by conventional magnetic and density techniques, and then selected by hand-picking under a binocular microscope. Representative zircon grains along with TEMORA standard (417 Ma) were embedded in epoxy resin and polished to expose the crystals for dating. Transmitted and reflected light micrographs as well as cathodoluminescence (CL) images were obtained for the polished zircon grains before U–Pb isotope analyses in order to reveal their internal structure and external morphology and guide the selection of potential analytical spots. The CL images were made using a

HITACHI S3000-N Scanning electron microscope at Beijing SHRIMP Center, Chinese Academy of Geological Sciences, Beijing.

Zircon from the diorite was dated in situ on an ArF-excimer (193-nm wavelength) laser ablation multiple-collector inductively coupled plasma mass spectrometer (LA-ICPMS) at the Tianjin Institute of Geology and Mineral Resources, China Geological Survey. The ICP-MS used was a Neptune made by Thermo Fisher, and the UP193-FX ArF laser ablation system (ESI Company) was used for the laser ablation experiments. The instrumental conditions and analytical processes were similar to those described by [Hou et al. \(2009\)](#page-13-0). U and Pb concentrations were calibrated by using TEMORA and GJ-1 as external standards [\(Jackson et al., 2004](#page-13-0)). $^{207}Pb/^{206}Pb$, $^{206}Pb/^{238}U$, $^{207}Pb/^{235}U$, and $^{208}Pb/^{232}Th$ ratios, calculated using the ICPMSDataCal program ([Liu et al., 2009\)](#page-13-0) and the Isoplot program ([Ludwig, 2003](#page-13-0)), were corrected for both instrumental mass bias and elemental and isotopic fractionation by using standard glass NIST612 as an external standard. The age data are in Supplementary Table 1.

In addition, in situ U–Th–Pb isotopic analyses of zircons from the alkali mafic dikes were carried out using the sensitive high resolution ion micro probe (SHRIMP IIe-MC) at Beijing SHRIMP Center, Chinese

Academy of Geological Sciences, Beijing. The analytical procedures are after [Song et al. \(2002\)](#page-13-0) and [Williams \(1998\)](#page-14-0). Primary beam size was 25 μm, and each site was rastered for 150–200 s prior to the analyses. Between each three analyses, a standard zircon (TEMORA) was analyzed for calibration. The common lead correction was referred to the measured 204Pb abundances. Data processing was performed using the SQUID and ISOPLOT programs [\(Ludwig, 2003](#page-13-0)). Errors for individual analyses are quoted at 2σ (two standard deviation) with 95% confidence level. The data of the ages are listed in Supplementary Table 2.

Hf isotope data was obtained using a Neptune Plus multiplecollector inductively coupled plasma mass spectrometer with RESOlution M-50-LR laser ablation (ArF-excimer 193-nm wavelength) at the Guangzhou Institute of Geochemistry of Chinese Academy of Sciences. The Hf isotopic analysis spots coincided with the U-Pb dating spots, and Penglai zircons were selected as reference material [\(X.H. Li et al.,](#page-13-0) [2010](#page-13-0)) with the precision being 0.282882 ± 0.000006 (2 s, N = 56). The analytical procedures and principles used were similar to those described by [Wu et al. \(2006\)](#page-14-0). In the experiment, helium was used as the abrasion material carrier gas, and the diameter of the laser beam spot is 33 μm with energy of 80 mJ and repeat frequency of 8 Hz. The Hf isotopic results are listed in Supplementary Table 3.

4.2. Electron microprobe analyses

Major element compositions of plagioclase and biotite in the diorite sample (Wjl070403) were measured by the electron probe micro analyzer (EPMA, JEOL JXA-8100) at the Second Institute of Oceanography, State Oceanic Administrator, Hangzhou, China. Quantitative analysis followed the analytical procedure of [Iizuka et al. \(2005\),](#page-13-0) using a 5 μm defocused beam with a beam current of 20 nA and acceleration voltage of 15 kV. The measured X-ray intensities were corrected by the ZAF method using the standard analysis of synthetic standard minerals with various diffracting crystals. The relative standard deviations (RSD) for Si, Na and K were less than 1 wt.%, and for other elements was less than 0.5 wt.%. Detection limits were less than 500 ppm for all elements based on 2σ of standard calibration. Concentrations of ferrous and ferric iron were calculated using stoichiometry and charge balance. More detailed information for this method is provided in [Y.Q. Li et al.](#page-13-0) [\(2012\)](#page-13-0), and the representative compositional data are given in Supplementary Tables 4 and 5.

4.3. Major and trace elemental analyses

Thirteen samples of alkali diabase, diorite and syenitic rocks were selected for major and trace elemental analyses. Major elements were analyzed by X-ray fluorescence (XRF) (Rigaku 3270E) at the Guangzhou Institute of Geochemistry, Chinese Academy of Sciences. Analytical precision was generally better than 2% for most oxides and better than 1% for SiO₂. Trace element data were obtained at Harvard University of USA by inductively coupled plasma mass spectrometer (ICP-MS) using the analytical procedure of [Bézos et al. \(2009\)](#page-13-0). ⁷²Ge, ¹⁰³Rh, ¹¹⁵In, ¹⁶⁹Tm and ²⁰⁹Bi were selected as internal standard normalization and international standards BHVO-2, BCR-2, AGV-1, DNC-1, W-2 and JB-2 were chosen as calibration standards for calculation of element concentrations of the measured samples. The analytical precision was better than 5% for trace elements. The major and trace elemental data from 13 samples and international standard materials in this paper are listed in [Table 2](#page-5-0) and Appendix 1.

4.4. Sr–Nd–Pb isotopic analyses

The samples selected for Sr–Nd–Pb isotopic analyses were ground with an agate mill, and powders were spiked with mixed isotope tracers, dissolved in Teflon capsules with $HF + HNO₃$ acid, and separated by conventional cation-exchange techniques with diluted HBr as an eluant. The isotopic measurements were performed on a Finnigan MAT-262 mass-spectrometer at the Institute of Geology and Geophysics, Chinese Academy of Sciences. The mass fractionation corrections for Sr and Nd isotopic ratios were based on ${}^{86}Sr/{}^{88}Sr = 0.1194$ and 146 Nd/ 144 Nd = 0.7219. Repeat analyses yielded a 87 Sr $/86$ Sr ratio of 0.710252 \pm 0.000025 for the NBS-987 Sr standard and a ¹⁴³Nd/¹⁴⁴Nd ratio of 0.512118 \pm 0.000012 for the JNdi-1 Nd standard. Repeat analyses yielded ²⁰⁴Pb/²⁰⁶Pb = 0.05897 \pm 0.00015, ²⁰⁷Pb/²⁰⁶Pb = 0.91445 ± 0.00080 , $^{208}Pb/^{206}Pb = 2.16170 \pm 0.00180$ for NBS981 Pb standard. Detailed descriptions of the analytical techniques are given by [Wei et al. \(2004\)](#page-14-0) and [Zeng et al. \(2014\)](#page-14-0). All analytical results in this study are presented in [Tables 3 and 4.](#page-6-0)

5. Results

5.1. Zircon U–Pb age and Hf isotopic results of the Wajilitag diorite

Zircon grains from the sample (wjl050710) of the diorite are mostly euhedral, transparent, colorless, and 100–300 μm in length, with length-to-width ratios of 1.5–3. Under CL images [\(Fig. 3\)](#page-7-0), most crystals have euhedral concentric zoning, consistent with a magmatic origin [\(Belousova et al., 2002](#page-13-0)). Analyses were conducted on 29 zircon grains, and the isotopic ratios and calculated ages are listed in [Table 2.](#page-5-0) In the $^{206}Pb/^{238}U-^{207}Pb/^{235}U$ concordia plot ([Fig. 4\)](#page-7-0), the 29 analyses are concordant within error, and yield a weighted mean $^{206}Pb/^{238}U$ age of 275.2 ± 1.2 Ma (2σ, MSWD = 0.30).

Zircons from the alkali mafic rock (WJL081907) are euhedral but partly fragmented. Six transparent, colorless and intact grains were selected for analyses, and the isotopic ratios and calculated ages are listed in Supplementary Table 2. In the $206Pb/238U$ vs. $207Pb/235U$ concordia plot

Table 2

Major, trace and rare earth elements of the Wajilitag alkali mafi^c rock, diorite and syenitic rocks.

LOI: loss on ignition.

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 $(^{147}\text{Sm}/^{144}\text{Nd})_{\text{CHUR}} = 0.1967$ ([Jacobsen and Wasserburg, 1980\)](#page-13-0); $(^{143}\text{Nd}/^{144}\text{Nd})_{\text{CHUR}} = 0.512638$ [\(Goldstein et al., 1984](#page-13-0)); Nd depleted mantle model ages (TbM) were calculated after the formula in [Goldstein et al. \(1984\)](#page-13-0) using (¹⁴⁷Sm/¹⁴⁴Nd)_{DM} = 0.2137 and (¹⁴³Nd/¹⁴⁴Nd)_{DM} = 0.51315 ([Peucat et al., 1989\)](#page-13-0) at the present day. $\lambda_{\rm Rb} = 1.42 \times 10^{-11}$ a⁻¹ ([Steiger and Jager,](#page-13-0) [1977\)](#page-13-0); $\lambda_{\rm sm} = 6.54 \times 10^{-12} \text{ a}^{-1}$ [\(Lugmair and Marti, 1978](#page-13-0)).

[\(Fig. 4](#page-7-0)), the 6 analytic spots were concordant within error, and yielded a weighted mean $\frac{206}{9}$ b/ $\frac{238}{1}$ U age of 281.4 \pm 1.7 Ma (20, MSWD = 2.1).

The Hf isotopic results of zircons in the diorite are listed in Supplementary Table 3. The initial ratios of ¹⁷⁶Hf/¹⁷⁷Hf are between 0.282588 and 0.282737 (with an average of 0.282669), and the calculated $\varepsilon Hf(t)$ values range from −0.9 to 4.4 (with an average of 2.0). The Hf depleted mantle model ages (T_{DM}^{Hf}) of diorite range between 733 and 927 Ma.

5.2. Mineral chemistry

A diorite sample (WJL070403) was selected for EPMA analyses [\(Fig. 5](#page-8-0) and Supplementary Tables 4 and 5). The compositions of zoned plagioclase in the diorite range from $An_{40}Ab_{57}Or_3$ in the core to $An_{35}Ab_{63}Or_2$ in the rim, in other words showing 'normal" zonation with decreasing anorthite content from core to rim. Biotite from the samples has high fluorine content (1.6–1.9 wt.%), X_{Mg} values $(Mg/[Mg + Fe^{2+}]$ of 0.73–0.76 and TiO₂ content of 5.0–5.8 wt.%, and low chlorine content (0.13–0.22 wt.%).

5.3. Whole-rock geochemistry

The Wajilitag diorite and syenitic rocks fall in the diorite to nepheline syenite region on the TAS diagram with $SiO₂$ contents of 52.0–57.3 wt.% [\(Fig. 6a](#page-8-0)). The aegirine–nepheline syenites and syenite porphyry plot in the peralkaline to weakly peraluminous fields ([Fig. 6](#page-8-0)b). On Harker dia-grams [\(Fig. 7](#page-9-0)), Fe₂O_{3t}, TiO₂, MgO and P₂O₅ are negatively correlated with $SiO₂$ content, suggesting the dominant role of fractional crystallization during the formation of these rocks. The decreasing trends of $TiO₂$ and P_2O_5 illustrate fractionation of ilmenite and apatite.

The alkali mafic rock, syenite porphyry and aegirine-nepheline syenite are rich in total alkalis (11.5–14.3 wt.%), Al_2O_3 (19.4–21.1 wt.%), Zr, Hf and Ba, and have higher $Na₂O/K₂O$ ratios (>1) and lower Mg# (15–35), and K, P and Ti contents. They have higher total rare earth element (REE) abundances (ranging from 308 to 736 ppm) than the diorite. The rocks also have high LREE/HREE (chondrite normalized) ratios (13.2–22.5) than the diorite ([Fig. 8](#page-10-0)a). The alkali mafic rocks and aegirine–nepheline syenite have negative Ta anomalies implying the rutile crystallization, in contrast to the absence of a Ta anomaly for the diorite and syenite porphyry. The significant negative anomalies of K, P and Ti in the alkali mafic rocks and syenitic rocks may be the result of alkali feldspar, apatite and titanite crystallization. The whole-rock geochemical characteristics of diorite samples are generally similar to the syenitic rocks, but some minor differences can be observed. For example, the diorite samples have higher contents of $SiO₂$, MgO, P₂O₅, CaO, $Fe₂O₃$, TiO₂, but not Al₂O₃ ([Fig. 7](#page-9-0)). The diorites have higher Mg# values (59–64) and lower alkali contents (8.3–9.7 wt.%) and total REE abundances. The diorite rocks show chondrite-normalized REE patterns that are consistently left-sloping, but the LREE/HREE ratios of the diorite rocks are consistently lower than for syenitic rocks ([Fig. 8a](#page-10-0)). On trace element spider diagrams ([Fig. 8b](#page-10-0)), the diorite samples do not show strongly enrichment or loss in LILE and HFSE. All rock samples lack Eu anomalies in [Fig. 8a](#page-10-0).

5.4. Whole-rock Sr–Nd–Pb isotopic compositions

The initial isotope ratios were calculated at $t = 275$ Ma. The samples of syenite porphyry and aegirine–nepheline syenite have variable Sm and Nd contents but relatively constant 147Sm/144Nd and 143Nd/144Nd ratios ranging from 0.0855 to 0.1006 and 0.51243 to 0.51248, respectively, corresponding to ε Nd(t) values of 2.9–4.1. The diorite samples have different ¹⁴⁷Sm/¹⁴⁴Nd and ¹⁴³Nd/¹⁴⁴Nd ratios ranging from 0.1051 to 0.1089 and from 0.51229 to 0.51233, respectively, corresponding to ε Nd(t) values of 0.09-0.78. The Nd depleted mantle model ages ($T_{\text{DM}}^{\text{Nd}}$) of syenitic rocks range from 611 to 692 Ma whereas the $T_{\text{DM}}^{\text{Nd}}$ of diorite samples are 913-943 Ma.

Among eleven samples analyzed, the diorites and syenitic rocks have lower ⁸⁷Rb/⁸⁶Sr ratios of 0.0808–0.3339, corresponding to the lower $(^{87}Sr/^{86}Sr)$ _i ratios of 0.70343-0.70583.

Pb isotopic ratios are also distinctive, with the ²⁰⁶Pb/²⁰⁴Pb ratios of the syenite, aegirine–nepheline syenite and diorite samples ranging from about 18.66 to 18.93, 19.29 to 19.51 and 18.03 to 18.14, respectively (Table 4).

Fig. 3. CL (cathodeluminescence) images of dated zircon crystals from the diorite (WJL050710) and alkali mafic rock (WJL081907) in the Wajilitag area. Note: The red circles, blue circles and numbers represent the analyzed spots for zircon U-Pb dating, Hf isotopes and $\varepsilon Hf(t)$, respectively.

6. Discussion

6.1. The timing of emplacement of the Wajilitag complex and its vanadium titano-magnetite mineralization

A U–Pb age of ca. 275 Ma for the youngest units of the complex and an age of ca. 281 Ma for the large-scale Fe–Ti oxides mineralized were determined in this study. Based on the field observation, the alkali mafic dikes intruded into the clinopyroxenite, and the latter contained the large Fe–Ti oxide deposit. The zircon U–Pb dating of alkali mafic dike yielded the age of 281.4 \pm 1.7 Ma, which confirms that the clinopyroxenite unit and its associated Fe–Ti oxide deposit formed earlier than ca. 281 Ma. Furthermore, the diorite body was located on the top of the complex, and the age of 275.2 ± 1.2 Ma obtained from the diorite may imply for the terminal age of the complex.

In the Tarim LIP, igneous activity occurred from ca. 300 Ma to 270 Ma [\(Table 1\)](#page-4-0) [\(Li, 2013; Li et al., 2011; Xu et al., 2014; D.Y. Zhang et al., 2013](#page-13-0)). It is noted that the basalts are regarded as one of the earliest units of the Tarim LIP [\(Z.L. Li et al., 2012; Tian et al., 2010; X. Yu et al., 2011;](#page-13-0) [Zhang et al., 2012](#page-13-0)) and the mafic–ultramafic layered intrusions, intermediate-felsic bodies, mafic dikes and bimodal dikes make up the younger part of the Tarim LIP [\(Li et al., 2011; Yang et al., 2007; Zhang](#page-13-0) [et al., 2008](#page-13-0)). From [Table 1](#page-4-0), it is apparent that the dikes of the Tarim LIP, were emplaced over an age range of ca. 284–272 Ma. The age of the alkali mafic dike is 281 Ma and provides a constraint on the timing of emplacement of the clinopyroxenite and associated Fe–Ti oxide mineralization.

It is noted that the emplacement age of the diorite is identical with a zircon SHRIMP U–Pb age of 272 \pm 1.2 Ma for a quartz syenite dike of Xiaohaizi area in the Bachu County [\(Yang et al., 2007\)](#page-14-0) and is similar to

Fig. 4. (a) LA–MC–ICPMS U–Pb zircon concordia diagrams for the diorite (WJL050710) and (b) SHRIMP U–Pb zircon concordia diagram for alkali mafic dike (WJL081907) belonging to the Wajilitag intrusive complex.

Fig. 5. (a) and (b), and (c) and (d) showing analyzed point locations of biotites and plagioclases from the diorite, respectively.

the zircon U–Pb age of 274 ± 2 Ma for a quartz syenite in the Wajilitag intrusive complex ([Zhang et al., 2008](#page-14-0)). Combined with other published U–Pb data, it can be deduced that ca. 275 Ma of the emplacement time of the Wajilitag intrusive complex matches the ages for bimodal dikes in Bachu County and possibly represents the terminal age of Wajilitag complex formation.

Based on the ages we obtained for the alkali mafic rock and diorite, it can be speculated that the duration of the Wajilitag intrusive complex magmatism is from at least 281 Ma to 275 Ma. In addition, the alkali mafic dikes were emplaced earlier than the diorite and syenitic rocks in the Wajilitag complex, and there are diverse periods of mafic dike emplacement in Bachu area.

6.2. Origin of the syenitic rocks and diorites

The negative correlations of MgO and $Fe₂O_{3T}$ vs. SiO₂ contents of the Tarim Permian intrusions most likely indicate fractional crystallization from a magma source. In Harker diagrams [\(Fig. 7](#page-9-0)), all Bachu intrusive rock samples show a clear trend, which reflects a strong fractional crystallization process. It is obvious that the intrusive rocks from ultramafic

Fig. 6. (a) SiO₂ versus K₂O + Na₂O classification diagram [\(Cox et al., 1979](#page-13-0)); (b) A/NK versus A/CNK plot showing the weakly perallaline to weakly peraluminous character of Wajilitag syenitic rocks ([Maniar and Piccoli, 1989](#page-13-0)). $A = Al_2O_3$, $N = Na_2O$, $K = K_2O$ and $C = CaO$.

to felsic in composition show significant fractional crystallization with a weak contribution from crustal assimilation ([Fig. 9](#page-11-0)). The alkali mafic rocks and syenitic rocks are rich in alkalis, and the alkalis in the diorite are relatively high, inferring that they formed in a within-plate environment. The alkali mafic rocks and syenitic rocks show enrichment of HFSE (e.g., Th, Nb, Zr and Hf) and depletion of K, P and Ti, implying fractional crystallization of alkali feldspar, apatite and ilmenite. In addition, trace element and REE patterns for the diorite are similar to the syenitic rocks, with both exhibiting no significant enrichment or loss of HFSE or LILE.

Biotite provides an important host for halogens. In natural biotites, the OH group may be completely replaced by fluorine, so the $F/(F + Cl + OH)$ value can be nearly 1 and the amount of chlorine substitution can be as low as $Cl/(F + Cl + OH)$ of < 0.1 [\(Munoz, 1984](#page-13-0)). Magnesium and ferrous contents in biotite are directly related to F–Cl–OH exchange between bio-tite and hydrothermal fluids [\(Munoz, 1984\)](#page-13-0). $F/(F + CI + OH)$ values (0.19–0.22) and high IV(F) and IV(F/Cl) values (Supplementary Table 3) from diorite samples indicate low water fugacity conditions. Therefore, the high temperature stability of biotite determined in water undersaturated conditions [\(Munoz, 1984](#page-13-0)) may not apply in this case. The samples have high fluorine contents (1.6–1.9 wt.%) with constant amounts of TiO₂ (5.0–5.8 wt.%) in biotite, which are consistent with relatively high temperature conditions inferred for biotite. Combined with the high $TiO₂$ and halogen content in biotite, we can speculate the environment for biotite formation may be under high temperature and low water fugacity conditions and a relatively deep depth in the crust.

The Wajilitag rocks that we have studied have low $(^{87}Sr/^{86}Sr)$ _i ratios ranging between 0.70343 and 0.70583 and positive ε Nd(t) values of 0.09–4.12. These samples fall in near the FOZO area [\(Hart et al., 1992;](#page-13-0) [Stracke et al., 2005](#page-13-0)) in the $(^{87}Sr/^{86}Sr)$ _i vs. $\varepsilonNd(t)$ diagram [\(Fig. 10](#page-11-0)), suggesting they may be derived from a plume or the degassed mantle. Among the diorite and syenitic rocks, there is a weak evolutionary trend from aegirine–nepheline syenite to diorite, which is probably due to fractional crystallization. Moreover, the ε Hf(t) values in zircon of basalts and intrusive rocks in Tarim LIP ([Fig. 11\)](#page-11-0) also show an evolutionary trend from an enriched mantle to depleted mantle source components. On the Pb isotopic graph [\(Fig. 12\)](#page-11-0), there is a trend from basalts and mafic–ultramafic rocks in enriched mantle source to syenitic rocks and diorite on the DMM-FOZO array. There is a tendency for the basalts from the Tarim LIP to be derived from an enriched mantle component

Fig. 7. (a) SiO₂ versus major elements and (b) SiO₂ versus trace element diagrams for the Tarim LIP rocks. Note: The data of mafic-ultramafic and felsic rocks are from [C.L. Zhang et al.](#page-14-0) [\(2010\)](#page-14-0), [Sun et al. \(2008\),](#page-14-0) [Y.Q. Li et al. \(2010\),](#page-13-0) [Yang et al. \(2007\)](#page-14-0), and [Zhang et al. \(2008\).](#page-14-0)

Fig. 7 (continued).

Fig. 8. (a) Chondrite normalized REE patterns, and (b) primitive mantle normalized spidergrams for the intermediate igneous rocks. The normalization values of primitive mantle and chondrite are from [Sun and McDonough \(1989\)](#page-13-0).

Fig. 9. ε Nd(t) versus SiO₂ for the Wajilitag complex rocks. Note: The fields for mafic rocks and felsic rocks are from [C.L. Zhang et al.](#page-14-0) [\(2010\)](#page-14-0), [Sun et al. \(2008\)](#page-14-0), [Yang et al. \(2007\)](#page-14-0), [Yu \(2009\),](#page-14-0) [Zhang et al. \(2008\)](#page-14-0), and [Zhou et al. \(2009\).](#page-14-0) Symbols are the same as in [Fig. 7.](#page-9-0)

whereas the Tarim intrusive rocks are from a FOZO-like mantle component ([Fig. 13\)](#page-12-0). We suggest that the Wajilitag complex may be mainly derived from plume mantle (FOZO-type), but may also have minor addition of an EMII-type OIB component which had some lithospheric mantle interacted beneath the Tarim Basin.

Combined with the previous studies of coeval basalts and intrusive rocks, this study of the geochemistry, Sr–Nd–Pb isotopes and zircon Hf isotopes of the Wajilitag syenitic rocks and diorite confirms that the Wajilitag intrusive rocks are derived mainly from a relatively depleted plume mantle source (FOZO-type) which assimilated recycled old enriched lithospheric mantle, and then underwent strong fractional crystallization within the crust.

6.3. Implication for the Tarim LIP evolution history

Diorite and syenitic rocks associated with LIPs are typically emplaced during the later part of the LIP event [\(De Waal and Armstrong, 2000;](#page-13-0) [Shellnutt et al., 2011; Song et al., 2008; Zhong et al., 2011\)](#page-13-0). In the Emeishan LIP, most syenites and A-type granites were derived by differentiation of basaltic magmas ([Shellnutt and Jahn, 2010; Shellnutt and](#page-13-0)

Fig. 10. ε Nd(t) vs. initial ⁸⁷Sr/⁸⁶Sr diagram for Wajilitag syenitic rocks and diorites. The fields of DMM, FOZO, EMI, EMII and HIMU are from [Hart \(1984\),](#page-13-0) [Hofmann \(2003\)](#page-13-0) and [Zindler and Hart \(1986\).](#page-14-0) The other data are from [C.L. Zhang et al. \(2010\)](#page-14-0), [X. Yu et al.](#page-14-0) [\(2011\)](#page-14-0), [Zhang et al. \(2008\)](#page-14-0), and [Zhou et al. \(2009\).](#page-14-0) Symbols are the same as in [Fig. 7](#page-9-0).

Fig. 11. The zircon ε Hf(t) values of the Group 1a basalt, Group 1b basalt and intrusive rocks in the Tarim LIP.

Note: the other data are from [Zhang et al. \(2008\),](#page-14-0) [D.Y. Zhang et al. \(2010\),](#page-14-0) [Zhang et al.](#page-14-0) [\(2012\)](#page-14-0), and [Li \(2013\).](#page-13-0)

[Zhou, 2007; Shellnutt et al., 2009a,b, 2012; Zhong et al., 2007, 2009,](#page-13-0) [2011\)](#page-13-0) or by mixing between crustal melts and primary basaltic magmas [\(Xu et al., 2008\)](#page-14-0). On the ε Nd(t) vs. (${}^{87}Sr/{}^{86}Sr$)_i isotope plot (Fig. 10), the rocks in this paper fall in near the FOZO area, showing the similar characteristics to the other Bachu Permian mafic–ultramafic rocks [\(Yang](#page-14-0)

Fig. 12. ²⁰⁶Pb/²⁰⁴Pb versus ²⁰⁸Pb/²⁰⁴Pb and ²⁰⁶Pb/²⁰⁴Pb versus ²⁰⁷Pb/²⁰⁴Pb plots for the Wajilitag syenitic rocks and diorites.

The fields of DMM, FOZO, EMI and EMII and Northern Hemisphere Reference Line (NHRL) are from [Zindler and Hart \(1986\)](#page-14-0), [Hart \(1984\),](#page-13-0) [Hofmann \(2003\)](#page-13-0) and [Stracke et al. \(2005\)](#page-13-0). The other plotted data are from [Zhou et al. \(2009\).](#page-14-0) Symbols are the same as in [Fig. 7.](#page-9-0)

Fig. 13. (a) ²⁰⁶Pb/²⁰⁴Pb versus ⁸⁷Sr/⁸⁶Sr and (b) ²⁰⁶Pb/²⁰⁴Pb versus ¹⁴³Nd/¹⁴⁴Nd plots for the Wajilitag syenitic rocks and diorites. The data sources are the same as in [Fig. 12](#page-11-0). Symbols are the same as in [Fig. 7.](#page-9-0)

[et al., 2007; Zhang et al., 2008](#page-14-0)). It's obvious that the Tarim LIP rocks have a significant trend followed a temporal evolution from the basalt to intrusive rocks. The Keping basalts have the highest $(^{87}Sr)^{86}Sr$)_i and lowest ε Nd(t) values indicating an enriched mantle source (dominated by an EMII component). As the later magmatic pulse of the Tarim LIP, the intrusive rocks in Bachu area show higher $εNd(t)$ values than the basalts, implying an injection of the more depleted mantle source components, probably a FOZO type plume mantle source.

On the other hand, zircon Hf isotopic data of Tarim LIP basalts and intrusive rocks [\(Li, 2013; Zhang et al., 2008, 2012\)](#page-13-0) indicate distinct magma sources ([Fig. 11](#page-11-0)). As seen in [Fig. 11,](#page-11-0) the basalts from Keping area have the lowest negative ε Hf(t) values, however, the intrusive rocks in the Wajilitag complex almost show the positive $\varepsilon Hf(t)$ values, which implies that the magma sources changed from enriched mantle to relatively depleted mantle through the protracted duration of the Tarim LIP. The Hf isotopes of the diorite are similar to the Hf isotopic results from the quartz syenite in the Wajilitag intrusion [\(Zhang et al., 2008](#page-14-0)). Both of them have positive zircon $\varepsilon Hf(t)$ values, but the ε Hf(t) values from the diorite range from -0.9 to 4.4 with an average of 2.0 which are lower than the ε Hf(t) values from the quartz syenite (5.5–8.9) in the Wajilitag complex ([Zhang et al., 2008](#page-14-0)) [\(Fig. 11](#page-11-0)). The difference of Hf isotopes between diorite and quartz syenite indicates an evolutionary trend from the diorite to quartz syenite.

Moreover, the $\mathsf{T}_{\mathsf{DM}}^{\mathsf{Hf}}$ of diorite range from 927 Ma to 733 Ma and are older than the model ages of 702-546 Ma for the quartz syenites [\(Zhang et al., 2008](#page-14-0)) and are younger than the model ages of 1200- 960 Ma for the basalts in the Keping area ([Li, 2013; D.Y. Zhang](#page-13-0) [et al., 2010; Zhang et al., 2012](#page-13-0)). Compared to the older Hf model ages of Keping basalts, the rocks in the Wajilitag complex show younger model ages, reflecting that the source of Tarim LIP originated from the EMII-type OIB mantle component to FOZO-like plume mantle.

Based on field occurrence, U–Pb age, petrochemistry and isotope characteristics mentioned above, combined with regional geology and magmatic evolutional history from previous studies ([Li, 2013; Li et al.,](#page-13-0) [2011; Z.L. Li et al., 2012; Yang et al., 2007; Zhang et al., 2008, 2012;](#page-13-0) [Zhou et al., 2009](#page-13-0)), our study suggests that the diorites of the Wajilitag complex may form through crystal fractionation of the mafic magmatism to become the later rock units of the Wajilitag complex and Tarim LIP, and the source of Wajilitag complex probably was FOZO-like plume mantle. In addition, the basalts and mafic–ultramafic intrusions in Tarim LIP, exhibit a regional source of this LIP changes from the enriched mantle (EMII type) to FOZO-like mantle plume source.

7. Conclusion

Based on the U-Pb ages, geochemical data, Sr–Nd–Pb isotopes and zircon Hf isotope analyses of the Wajilitag alkali mafic dikes, diorite and syenitic rocks, we summarized as follows.

- (1) The zircon U–Pb ages of the alkali mafic dike and diorite in the Wajilitag complex are 281.4 \pm 1.7 Ma and 275.2 \pm 1.2 Ma, respectively. It can be deduced that the ages of ca. 275 Ma and ca. 281 Ma indicate the terminal emplacement time of the Wajilitag intrusive complex and Fe–Ti oxides mineralization time, respectively. The former is consistent with previously published age constraints on mafic dikes and bimodal dikes in the Bachu area.
- (2) The higher fluorine and $TiO₂$ contents and low chlorine contents in biotite of the diorite in the Wajilitag complex indicate that the biotite formed under high temperature and low water fugacity conditions and a deep depth in the crust.
- (3) The evidence of lower (${}^{87}Sr/{}^{86}Sr$)_i, and positive high $\varepsilon Hf(t)$ and ϵ Nd(t) from the Wajilitag complex in this study suggests a depleted source (perhaps FOZO) reflecting a plume mantle source with minor contamination by an enriched component (dominated by EMII). Some evolved rocks (e.g., diorites) also experienced fractional crystallization from initially derived basaltic magma. In addition, geochemistry, Sr-Nd-Pb isotopes and Nd depleted mantle model ages indicate a magmatic evolutionary trend from the diorite to syenitic rocks.
- (4) The zircon U–Pb age and geochemistry of the newly discovered alkali mafic dike which intruded into the clinopyroxenite, indicate the formation time of the clinopyroxenite and Fe–Ti oxide mineralization could be earlier than 281 Ma. Compared with the ages for mafic dikes and bimodal dikes in Bachu area, it can be concluded that the dikes formed over a wide time range from 284 to 272 Ma.
- (5) Combined with the previous study of Permian different rock types in Tarim Basin, it can be deduced (based on published data) that the Wajilitag intrusive complex is regarded to belong to a younger pulse of the Permian Tarim LIP. Along with Early Permian basalts erupted and then mafic–ultramafic to felsic rocks intruded in Tarim Basin, the Tarim LIP rocks were derived from mantle source which changed from the enriched mantle component (dominated by EMII) mainly for the basalts to a depleted source (similar to FOZO-type mantle plume) for intrusive rocks, followed by strong fractional crystallization in crustal magma chambers to produce the more evolved phases (i.e., diorite and syenites).

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