The Paleoproterozoic Chibaisong Mafic-Ultramafic Intrusion and Cu-Ni Deposit, North China Craton: SHRIMP Zircon U-Pb and Re-Os Geochronology and Geodynamic Implications

Han Chun-Ming**, Wu Fu-Yuan¹, Xiao Wen-Jiao¹, Zhao Guo-Chun², Ao Song-Jian¹, Zhang Ji-Een¹, Wan Bo¹, Qu Wen-Jun² and Du An-Dao¹

¹Key Laboratory of Mineral Resources, Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing 100029, China
²Department of Earth Sciences, The University of Hong Kong, Pokfulam Road, Hong Kong, China
³National Research Center of Geoanalysis, Beijing, 100037, China

Abstract

The Chibaisong mafic-Ni–Cu sulfide deposit (Jilin Province, NE China) is located in the Jiao-Liao-Ji Belt of the North China Craton. The ore-bearing mafic-ultramafic intrusions intrude the metamorphic rocks of the lowermost Sidaolazi Formation of the Anshan Group. SHRIMP zircon U-Pb dating gives a maximum depositional age of 2188 ± 8 Ma (95% confidence level, MSWD=3.2, n=6) for the host sedimentary rocks of the Chibaisong deposits. Rhenium and osmium isotopic analyses of Ni-and Cu-bearing sulfide minerals from the deposit have been used to determine the timing of mineralization; the source of osmium, and, by inference, the source of ore metals. Sulfide ore samples have Os and Re concentrations ranging from 19 to 490 ppb and from 0.47 to 13.97 ppb, respectively. Analyses of these data yield a six-point isochron age of 1885 ± 94 Ma. An initial 187Os/188Os ratio of 0.80 ± 0.16 (mean square of weighted deviates=0.17) and ε²⁸S values of -1.1 to +0.7‰ for the ores, as well as initial 1⁸⁷Sr/1⁸⁶Sr ratios of 0.703-0.708 for mafilferrous intrusions, suggest a magmatic source for the ores, with the melts dominated by mantle contributions. The Chibaisong intrusion was previously considered to have formed under a post-orogenic extensional setting in the Early Cretaceous. However, our new Re-Os geochronological result indicates that the Chibaisong mafic-ultramafic intrusion, along with the Cu-Ni deposit, was related to a Paleoproterozoic extensional event, not resulting from an Early Cretaceous post-orogenic extension as previously considered.

Keywords: Re-Os isotopic dating; Cu-Ni deposit; Chibaisong; North China Craton

Introduction

Observations of the spatial and temporal setting of magmatic nickel sulfide deposits (or camps comprised of multiple deposits) indicate an empirical relationship with large lithospheric boundaries, and more specifically, to craton margins [1]. This relationship remains poorly documented in the scientific literature. The North China Craton (NCC) is the largest nickel producing area in China (Figure 1). Until now, two mines have been in production, and numerous other deposits have been found along the margin of the NCC. Since the discovery of the Jinchuan deposit in 1979, extensive geological exploration has been carried out in the NCC region, which led to the recognition of the NCC as one of the largest nickel-copper provinces in China. Over the last few years, numerous attempts have been made to directly or indirectly determine the ages of the nickel-copper provinces [2-5]. However, the timing of the magmatic and mineralization events and their correlations within the complexes remain unclear.

In this study, we present new U-Pb zircon and Re-Os isotopic data for the mineralized ultramafic body from the eastern copper-nickel belt in the Jiao-Liao-Ji Belt of the NCC in order to constrain the timing and origin of the mineralization. These new data provide important insights into understanding the mineralization processes and geodynamic environment of the Chibaisong Ni-Cu ore deposit. In addition, an attempt was also made to understand the provenance and geodynamic environment of the NCC copper-nickel province. An understanding of the mineralizing processes and the geodynamics is beneficial for Ni-Cu exploration on other mafic-ultramafic bodies in the region and throughout the NCC.

Regional Geology

The NCC is bound to the north by the Central Asian Orogenic Belt (CAOB) [6,7] and to the south by the Paleozoic to Triassic Qinling-Dabie-Sulu orogenic belt [8,9]. The CAOB underwent north-south directed subduction, and several episodes of collisions at the Solonker suture during the Palaeozoic and accretionary orogenesis from the late Proterozoic to the Mesozoic [6,7,10]. To the south, the Qinling-Dabie-Sulu orogenic belt formed through the continental collision between the NCC and the Yangtze Craton in the Triassic [8] (Figure 1).

The Archean to Paleoproterozoic basement of the NCC has been divided into four microcontinental blocks, namely the Yinshan, Ordos, Longgang and Nangrim Blocks (Figure 1), of which the Yinshan and Ordos Blocks are considered to have amalgamated along the EW-trending Khondalite Belt to form the Western Block at 1.95-1.92 Ga [11-14], the Longgang and Nangrim Blocks amalgamated along the Jiao-Liao-Ji Belt to form the Eastern Block at ~1.90 Ga [15-21], and finally, the Western and Eastern Blocks collided along the Trans-North China Orogen to form the coherent basement of the North China Craton at ~1.85 Ga [22-26].

The Chibaisong ultramafic-mafic intrusion and associated Ni-
Cu ore deposit are located in the Paleoproterozoic Jiao-Liao-Ji Belt in the Eastern Block of the NCC, which consists predominantly of Paleoproterozoic metamorphic basement rocks, overlain by Paleozoic and Mesozoic sedimentary cover (Figure 2). The Archean basement rocks are mainly composed of supracrustals (Anshan Group) and tonalitic-granodioritic gneisses metamorphosed in granulite- and amphibolite-facies, with minor two mica granite and hypersthene granite [27]. The Paleoproterozoic rocks are dominated by supracrustal rocks, named the Ji’an and Laoling Groups, and minor ultramafic, mafic and granitic intrusions and dykes [27].

Local geology

The host rocks of the Chibaisong Cu-Ni deposit are metamorphic rocks of the Sidaolazi Formation of the Archean Anshan Group, which consists predominantly of biotite-amphibole-plagioclase gneiss, migmatite and amphibolite, all of which show strong ductile deformation (Figure 3). The Chibaisong intrusions, extending 4800 m along strike, with a width between 40 and 140 m (Figure 3), and a depth of 1000 m (Figure 4), are composed mainly of diabase gabbro, livinenerite-gabbro, plagioclase herzolite, norite-gabbro and gabbro-porphyrityte [28]. The Cu-Ni sulfide ores are hosted in the plagioclase lherzolite and norite-gabбро units. The orientation of the orebody is almost consistent with that of the intrusion, with its northern part striking 5-10° and dips to the SEE at 55 to 86°, and its southern part dipping to the NWW at 63 to 85° [28]. The orebody occurs as a tabular or bedlike unit, with a maximum length up to 200 m and a thickness of up to 40 m at the surface. The exploratory profile section reveals that the ore body extends as much as 1000m down-dip.

Ore minerals are dominantly pyrrhotite, chalcopyrite, pentlandite, violarite, pyrite and magnetite. Associated noble metal-bearing minerals are melonite, michenerite, bitepalladite, sperrylite, hessite and electrum. Gangue minerals are mainly olivine, pyroxene, plagioclase, hornblende, biotite, chlorite and serpentine.

Ore minerals are characterized by euhedral, subhedral, anhedral, subhedral-anhedral, porphyritic, corrosion, relict-replacement, reaction rim, pseudomorphic and skeletal crystal textures. Ore structures are mainly massive, vein, veinlet-disseminated, veinlet-mesh, or sparsely disseminated. The mineralization shows a circular zoning pattern, from high-grade massive ore in the center outwards to disseminated ores, gradually changing into sub-econonomic ores.

Wall-rock alteration related to mineralization in the Chibaisong Cu-Ni deposit includes chloritization, sericitization, carbonatization, serpentinization. Characteristic minerals in the alteration zones are chlorite, serpentine, sericite and carbonate.

Sampling and Analytical Techniques

Sampling

Sample collection: A fresh 40 kg olivine-norite-gabbro specimen used to obtain SHRIMP zircon U-Pb ages was collected in the eastern part of No.1 ore body, with coordinates of 125°42’38”E, 41°40’07”N.

For Re-Os dating, we collected 6 samples from fresh open-pit mining faces of the Chibaisong deposit. Samples were collected at a depth of 300 m in the eastern part of ore body (Figure 3).
Sample selection: Zircon grains from sample Y041 were separated at the Laboratory of the Institute of Regional Geology and Mineral Resources Survey in Hebei Province. The gneiss sample was jaw-crushed, split, and ground to 100 μm using conventional methods, then primarily separated using electromagnetic and heavy liquid techniques. Finally, grains were handpicked according to their euhedral shapes and transparency, using a binocular microscope. Zircons were separated from the crushed samples using heavy liquids and a Frantz magnetic separator. Approximately 80 zircons from sample Y041 were handpicked, mounted in epoxy resin and polished until the grain centers were exposed.

Single sulfide grains were also selected at the above-mentioned laboratory. Crude ore samples were jaw-crushed, elutriated by water, separated by Frantz magnetic techniques, panned by alcohol, and finally hand sorted under the microscope. The weight of six pure pyrrhotite samples was about 5g each. To avoid Re contamination from steel and tungsten carbide crushing equipment, the pure pyrrhotite powders from the Fujia deposit were pulverized to b200-mesh grain size using a ceramic jaw crusher and agate mill. The purity of the samples was greater than 99% prior to Re–Os isotopic analyses.

Analytical procedures

Cathodoluminescence (CL) images: The CL images were obtained using a CAMECA SX50 microprobe at the Institute of Geology and Geophysics, Chinese Academy of Sciences in Beijing, in order to identify internal textures and choose potential target sites for later U–Pb analyses. The working conditions during CL imaging were at 15 kV.

SHRIMP U–Pb isotope analytical techniques: Zircon-U-Th-Pb analyses were performed using the Sensitive High-Resolution Ion Microprobe (SHRIMP II) at the Beijing SHRIMP Laboratory, Chinese Academy of Geological Sciences. Details of the analytical methods and procedures for zircon dating using SHRIMP have been described by [29-32]. Analytical target sites within zircons were chosen for SHRIMP U-Pb zircon dating. Due to small amounts of 207Pb formed in young (i.e., ~1000 Ma) zircons, which results in low count rates and high analytical uncertainties, the determination of the ages for young zircons has to be based primarily on their 206Pb/238U ratios; older zircon ages are derived from 206Pb/207Pb ratios [33]. During the SHRIMP analyses, the intensity of the preliminary ion current was 2.5-4.5 nA and spot size averaged 25-30 μm, using peakjumpingscanningmode. The measured 206Pb/238U ratios were used for inter-element fractionation corrections with TEMORA zircon standards (417 Ma; 206Pb/238U=0.06683). The SL 13 zircon standard was used for calibration of U, Th and Pb contents. Common lead corrections were based on measured 206Pb assuming that the 208Pb/232U-208Pb/238U age was accordant. The standard sample was measured after every three points, in order to insure the reliability of the measured results and then to monitor the stability of the instrument. The isotopic ratio and element content data were handled using ISOPLOT programs [34]. The age uncertainties are cited as 1σ, and the weighted average ages of 206Pb/238U used have a confidence level (2σ) of 95%.

Re–Os isotope analytical technique

Re-Os isotopic analyses were performed at the National Research Institute.

Figure 2: Geological map of the eastern part of Jilin province [27].

Figure 3: Geological and showing sampling locations map No. 1 ore body of the Chibaisong Cu-Ni deposit [28].
Center of Geoanalysis. The details of the chemical procedure are described by [35-37], and are briefly summarized as follows.

Enriched $^{186}$Os and enriched $^{187}$Re were obtained from the Oak Ridge National Laboratory, USA. A Carius tube (a thick-walled borosilicate glass ampoule) digestion was used. The weighed sample was loaded in the Carius tube through a thin necked long funnel. The mixed $^{186}$Os and $^{187}$Re spike solutions and 2 ml of 12M HCl and 6 ml of 15 M HNO$_3$ were loaded while the bottom part of the tube was frozen at -80°C to -50°C in an ethanol-liquid nitrogen slush; the top was then sealed using an oxygen-propane torch. The tube was then placed in a stainless-steel jacket and heated for 24 hours at 230°C. Upon cooling, the bottom part of the tube was refrozen, the neck of the tube was broken, and the contents of the tube were poured into a distillation flask and the residue was washed out with 40 ml of water.

Osmium was distilled at 105-110°C for 50 minutes and trapped in 10 ml of water. The residual Re-bearing solution was saved in a 150 ml Teflon beaker for late Re separation. The water trap solution was used for ICP-MS (TJA X-series) determination of the Os isotope ratio. Cation-exchange resin was used to remove Na when the salinity of the Re-bearing solution was more than 1 mg/ml [38].

The ICP-MS analysis was conducted on a TJA PQ ExCELL ICP mass spectrometer. The instrument was optimized to: $>5 \times 10^4$ cps for 1 ng ml$^{-1}$ In and $>5 \times 10^4$ cps for 1 ng ml$^{-1}$ U. Data acquisition was performed in peak-jumping mode, 3 points/µl; dwell time=15 ms/point and number of scan=200 for 5 ppb of Re in solution. The reproducibility by ICP-MS is 0.3% (RSD, 2S, n=5); by using water as an absorbent for OsO$_4$, the sensitivity of Os by ICP-MS increases significantly. For 0.2 ppb of Os solution, the reproducibility is 0.3% (RSD, 2S, n=5).

If a minor $^{186}$Os signal was observed when measuring Re, the $^{187}$Re signal was appropriately corrected for $^{187}$Os using the $^{186}$Os/$^{187}$Os ratio of the spiked Os solution. Conversely, if a minor $^{187}$Re signal was observed while analyzing the Os-bearing solution, $^{187}$Os was appropriately corrected for $^{187}$Re using the measured $^{185}$Re/$^{187}$Re of the spiked sample. The corrections were generally minor and constituted no more than 0.1% of the isotope signal. The maximum correction percentage that we used was always less than 1%.

The mass fractionation can be corrected using an interlaboratory isotope reference standard. Using the $\lambda_{238}$U value of [39] and $\lambda_{235}$ value of [40], a value for $\lambda_{187}$Re of $1.6689 \pm 0.0031 \times 10^{-11}$a$^{-1}$ was determined. This value is nominally higher (ca. 0.1% and ca. 0.2%) than the value determined by [40], but is within the calculated uncertainty. So, we used the $\lambda_{187}$Re$1.666 \pm 0.005 \times 10^{-11}$a$^{-1}$ determined by [40].
Average blanks for the total Carius tube procedure as described above were ca. 10 pg Re and ca. 0.1 pg Os. 187Os was not detected. Three reference materials were used to check the analytical results.

The uncertainty in each individual age determination was about 1.02% including the uncertainty of the decay constant of 187Re, uncertainty in isotope ratio measurement, and spike calibrations. The decay constant used for 187Re of 1.666×10^{-11}a^{-1} has an absolute uncertainty of ± 0.0017 (1.0%) [40].

**Results**

**SHRIMP U–Pb dating results**

Twelve analyses of 12 grains from Chibaisong olivine-norite-gabbro were analyzed on the CZ3 standard (Figure 5). Of these analyses (Table 1), two age populations are identified (Figure 6), of which one age group consists of eight grains gave a Th/U ratio larger than 4.0, (Table 1), two age populations are identified (Figure 6), of which one gabbro were analyzed on the CZ3 standard (Figure 5). Of these analyses above were ca. 10 pg Re and ca. 0.1 pg Os. 187Os was not detected. Three zircons and are thus regarded to be detrital zircons that suffered and Th/U ratios ranging respectively from 84 to 393 ppm, 175 to 823 ppm and 0.33 to 0.48, which are different from typical metamorphic zircons and are thus regarded to be detrital zircons that suffered variable Pb loss in metamorphism. Thus, the concordant age of 2188 ± 8 Ma may be the best constraint for estimating the maximum age of the Chibaisong deposit.

**Re–Os dating results**

The abundance of Re and Os and the osmium isotopic compositions of the Cu-Ni sulfide ores from the Chibaisong mine are shown in Table 2. Re and Os concentrations for the massive sulfide of the Chibaisong deposit are high, ranging from 0.47 to 13.97 ppb Os and 19 to 490 ppb Re (Table 2). The analyses on six samples define a Re-Os isochron with an age of 1885 ± 94 Ma, with initial 187Re/187Os ratio of 0.80 ± 0.16 and a mean square of weighted deviation (MSWD) of 0.17 (Figure 7). The isochron age was calculated by means of the 187Re decay constant of 1.666×10^{-11}year [40] using the ISOPLOT software (Model 3; [34]). This isochron age can reflect the ore-forming age of the Chibaisong Cu-Ni deposit. The combination of high Os concentrations coupled with low Re/Os ratios is not typical of most low-tenor, basalt hosted magmatic sulfides (e.g. Duluth Complex), which exhibit much lower Os concentrations, coupled with higher Re/Os ratios [41]. This suggests a parental magma for the Chibaisong sulfides that had both a higher Os concentration and a lower Re/Os ratio than a typical basaltic magma.

**Discussion**

**Initial 187Os/188Os and source of ore-forming metals**

The Re-Os isotope system has been recognized as a geochemical tool not only for directly dating mineralization but also for defining the ore forming process of Cu-Ni sulfide deposits, and thus is a powerful tracer of sulfide ore formation and can be a highly sensitive monitor of the extent of crustal involvement during ore genesis [42,43]. Since the initial 187Os/188Os ratios of the crust (0.2-10) are higher than those of the mantle (0.11-0.15, [44]), 187Os/188Os ratios can be used to readily discern different sources [45].

The Chibaisong Cu-Ni sulfides ores possess an initial 187Os/188Os ratio of 0.80 ± 0.16 (Figure 7), which is considerably greater than chondritic or value those of the mantle 187Os/188Os ratio at 1885 Ma [35,46,47], indicating that crustal components were involved in the Os source of the Chibaisong ores.

In order to describe the Os isotopic composition at a given time, a parameter of γOs was introduced by [46-48].

\[ γ_{Os}(T)=100 \left(\frac{187Os/188Os_{sample}(T)}{187Os/188Os_{chondrite}(T)}-1\right) \]

Owing to high Re/Os ratios in the crust, γOs will have a large positive value with increasing crustal material entering the magmatic or ore-forming systems, and, by contrast, Re-loss in the systems can cause

| Spot  | U (μg/g) | Th (μg/g) | Pb (μg/g) | Th/U |
|-------|---------|---------|---------|------|
| Y041-1| 411.80  | 2797.9  | 142.53  | 6.79 |
| Y041-2| 864.48  | 4227.2  | 300.92  | 4.89 |
| Y041-3| 823.65  | 393.17  | 194.20  | 0.48 |
| Y041-4| 397.54  | 132.28  | 116.96  | 0.33 |
| Y041-5| 562.49  | 230.91  | 145.42  | 0.41 |
| Y041-6| 692.64  | 4968.8  | 529.77  | 7.17 |
| Y041-7| 410.28  | 2135.6  | 148.54  | 5.21 |
| Y041-8| 175.92  | 84.52   | 58.84   | 0.51 |
| Y041-9| 1025.2  | 8095.2  | 353.31  | 7.90 |
| Y041-10| 348.38 | 2169.1 | 123.73  | 6.23 |
| Y041-11| 746.13 | 7474.5 | 249.67  | 10.0 |
| Y041-12| 1126.3 | 14884 | 395.32  | 13.2 |

Table 1: SHRIMP zircon U–Pb analytical data of olivine-norite-gabbro from the Chibaisong intrusions.
The calculation formula is $\gamma_{187}$Re = 100[$^{187}$Os/$^{188}$Os] sample (T)/($^{187}$Os/$^{188}$Os) chondrite (T)-1, where ($^{187}$Os/$^{188}$Os) chondrite=0.09531+0.40186 ($e^{(87\text{Os}/86\text{Sr})_{chondrite}}$-1). This implies that ores and melts at the Chibaisong deposit were all dominated by mantle sources, although some minor amount of crustal material may have been assimilated into the melts.

### Age of mineralization and its geodynamic significance

The Chibaisong intrusion was previously considered to have been linked to a post-orogenic extensional setting in the Early Cretaceous [5]. However, our new SHRIMP and Re-Os geochronological results indicate that the Chibaisong mafic-ultramafic intrusion, along with the Cu-Ni deposit, was probably related to the Paleoproterozoic mantle plume or superplume, rather than in the Early Cretaceous post-orogenic extension setting.

This new SHRIMP and Re-Os age (1885 ± 94 Ma) for sulfide mineralisation at the base of the Chibaisong intrusion is marginally younger than the previously determined ages of the nearby Chibaisong intrusion (2188 ± 84 Ma). Working on the geochronology of dyke swarms in the NCC, Peng et al. [53] identified four dyke suites with SHRIMP...
U-Pb ages of 2147 ± 5, 1929 ± 8, 1834 ± 5 and 1778 ± 3 Ma. On the basis of this geochronology [52], Peng et al. [33] suggested that the older group of dyke suites could have been related to the amalgamation of the NCC to the Columbia supercontinent, whereas the younger suites were related to the breakup of Columbia and a mantle plume event at 2147-1778 Ma [52]. Mafic dykes in the Chibaisong region, have SHRIMP U-Pb and LA-ICP-MS ages of 2188 ± 8 and 2145 ± 19 Ma, respectively, confirming the Paleoproterozoic rifting processes within the NCC. The Chibaisong ultramafic-mafic intrusion was located within the Paleoproterozoic Jiao-Liao-Ji Belt which is considered as an intercontinental rift zone that developed about 2.2 Ga and the rifting basin was closed at about 1.93-1.90 Ga, followed by a post-orogenic extensional event at 1.88-1.85 Ga [15-17,53,54]. The Chibaisong ultramafic-mafic intrusion was likely the earliest product of this post-orogenic extensional event.

Conclusions

i. Zircon SHRIMP U–Pb ages of the Chibaisong magmatic Ni–Cu sulfide deposits indicate a maximum depositional age of 2188 ± 8 Ma. This result is consistent with the geological fact that ore-bearing mafic-ultramafic intrusions intruded into the Sidaolazi Formation of the Anshan Group.

ii. Six massive Ni–Cu sulfide ores of the Chibaisong deposit yielded a Re-Os isotopic isochron age of 1885 ± 94Ma, indicating that the ore-forming age was Paleoproterozoic.

iii. Massive Ni–Cu sulfide ores of initial 187Re/184Os ratio of 0.80 ± 0.16, and their yOs values ranged from +24 to +62, indicating that ore-forming materials came mainly from the mantle with crustal Os contamination, suggesting a mixture of mantle and crustal sources.

iv. The Chibaisong Cu-Ni deposit was located within the Paleoproterozoic Jiao-Liao-Ji Belt which was the earliest product of this post-orogenic extensional event.

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