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# Article The Relationships between Greenstone Belts and the Kryvyi Rih–Kremenchuk Basin in the Middle Dnieper Domain of the Ukrainian Shield Revealed by Detrital Zircon

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Abstract: Detrital zircons from two samples of metasandstones from the Lykhmanivka Syncline, Middle Dnieper Domain of the Ukrainian Shield (Skelevate Formation of the Kryvyi Rih Group), have been dated by the LA-ICP-MS U-Pb method. Metasandstones from the northern part of the syncline yield zircons belonging to four age groups:  $3201 \pm 12$  Ma,  $3089 \pm 11$  Ma,  $2939 \pm 8$  Ma, and  $2059 \pm 4$  Ma. All three Archean groups originated from similar rock types that crystallized at different times from the same mafic source (lower crust) with a  $^{176}Lu/^{177}Hf$  ratio of about 0.020. In contrast, zircon from metasediments from the southern end of the Lykhmanivka Syncline fall within two age groups:  $3174 \pm 13$  Ma, and  $2038 \pm 9$  Ma. In terms of Hf isotope compositions, the detrital zircons from the two oldest age groups in both samples are very similar. The source area was dominated by rocks of the Auly Group (3.27–3.18 Ga) and the Sura Complex (3.17–2.94 Ga). The proportion of zircons dated at 2.07–2.03 Ga, which reflects the timing of metamorphism, is 5%. The metamorphic nature of the Paleoproterozoic zircon allows us to define the maximum depositional age of the metasandstones of the Lykhmanivka Syncline at ca. 2.9 Ga, which is in good agreement with the earlier results from the metaterrigenous rocks of the Kryvyi Rih-Kremenchuk Basin. Our data also indicate the local nature of sedimentation and the absence of significant transport and mixing of detrital material within the basin.

**Keywords:** Greenstone belts; Kryvyi Rih–Kremenchuk Basin; Middle Dnieper Domain; Ukrainian Shield; Archean; metasandstone; detrital zircon

# 1. Introduction

Banded iron formations (BIFs), a major source of iron ore for the modern steel industry, are of significant economic value. They are quite common in Precambrian sedimentary successions and are important for understanding the Precambrian evolution of the atmosphere and the hydrosphere [1,2]. Moreover, it has been suggested that the deposition of major BIFs was coeval with episodes of emplacement and eruptions of large masses of mafic igneous rocks [3]. Indeed, in the Ukrainian Shield, as in many other places worldwide, BIF deposits are closely associated with greenstone belts and are repeatedly interbedded with mafic and felsic volcanic rocks [4,5]. In such cases, the age determination of BIFs is



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**Copyright:** © 2024 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). relatively straightforward and can be achieved through the dating of interbedded volcanic rocks. However, in the case of the Kryvyi Rih–Kremenchuk Basin, this approach is not suitable as the ca. 4 km-thick sedimentary sequence does not contain any volcanic rocks, except the metabasaltic Novokryvorizka Formation that occurs at the base of the succession. Metabasaltic rocks generally lack syngenetic minerals suitable for U-Pb dating, such as zircon and baddeleyite. In many cases, they may contain xenocrystic zircon captured on the way to the place of final crystallization or picked up at the source region [6–8], which complicates the interpretation of the obtained geochronological data.

Determining the depositional age of the sediments that fill the Kryvyi Rih–Kremenchuk Basin is one of the major geological issues in the Ukrainian Shield. According to the current regional stratigraphic chart of the early Precambrian of the Ukrainian Shield [9], the Kryvyi Rih Group is considered Paleoproterozoic. However, this age attribution is based solely on general considerations, such as its low degree of metamorphism and the presence of amphibolitized mafic dykes that yielded Proterozoic K-Ar ages. Only since the 2010s has geochronological data been collected in this basin [10–15] that indicated its Mesoarchean to Neoarchean age.

The Kryvyi Rih–Kremenchuk Basin is a narrow (up to 15 km wide), sedimentary basin that extends for over 170 km in an N–S direction. It is located in the central part of the Ukrainian Shield, within the Middle Dnieper Domain, and hosts some 35–40 Gt of iron ore. This basin has several small tail-shaped syncline structures of northwestern strike, e.g., Vysokopillya, East Hannivka, Zhovta Richka, which in turn connect with the Mesoarchean greenstone structures of the inner parts of the Middle Dnieper Domain (Figure 1). Such a spatial arrangement emphasizes the affinity of the Kryvyi Rih–Kremenchuk Basin with greenstone structures [4,16]. The basin is filled with metavolcanic and metasedimentary rocks of the Kryvyi Rih Group and the Hleyuvatka Formation (Figure 2).

The Kryvyi Rih Group includes four formations, from bottom to top:

- The lowermost Novokryvorizka Formation comprises metabasalts, amphibole schists, metaconglomerates, and metasandstones.
- The Skelevate Formation comprises metaterrigenous rocks (sandstones, conglomerates, schists) and a horizon of talc-rich schists. The thickness of the formation is up to 300 m.
- The Saksahan Formation is 650–800 m thick and represents the main productive BIF unit. It is composed of BIFs, separated by several horizons of schists and barren quartzites.
- The Hdantsivka Formation, up to 1100 m thick, is composed of schists, marbles, dolostones, sandstones, and ferruginous–siliceous rocks (BIFs). It rests disconformably on the Saksahan Formation.

The Hleyuvatka Formation comprises the upper part of the sedimentary succession, is up to 1800 m thick, and unconformably overlies the Kryvyi Rih Group. It is composed of metaterrigenous rocks including conglomerates, sandstones, and shales.

There have been several attempts to define the age of the rocks of the Kryvyi Rih– Kremenchuk Basin (Figures 1 and 2). The maximum depositional age of the metaterrigenous rocks of the Lativka Horizon that occur at the base of the Novokryvorizka Formation was defined at 3.0 Ga [10], whereas the age of an overlying metabasalt was established at 2.83–2.80 Ga [11]. Detrital zircon and monazite from metaterrigenous rocks of the Skelevate Formation yielded a maximum depositional age of 2.85 Ga [12,13,17] (Figures 1 and 2). The East Hannivka Belt, which represents the northernmost extension of the Kryvyi Rih Basin, has yielded a maximum depositional age of ca. 3.05 Ga [14]. The maximum depositional age of detrital zircon from quartzites of the Rodionivka Formation of the Inhul-Inhulets Group (part of the Zhovte Structure in the Pravoberezhna area) was defined at 2.68 Ga [15]. No U-Pb detrital zircon data have been obtained from the metaterrigenous rocks of the Skelevate Formation in the Lykhmanivka Syncline in the southern part of the Kryvyi Rih region.



**Figure 1.** (**A**) Simplified tectonic map of the Ukrainian Shield, modified after the tectonic map of the basement of the Ukrainian Shield [18]. (**B**) Schematic geological map of the Middle Dnieper Domain of the Ukrainian Shield. I—Kryvyi Rih Basin; II—Lykhmanivka Syncline; III—Vysokopillya greenstone belt; IV—Verkhivtseve greenstone belt; V—Zhovta Richka structure; VI—East Hannivka Syncline. The locations of the drill holes sampled for detrital zircon dating are indicated. For the approximate ages of various stratigraphic units and intrusive complexes, see Figure 2. Sampling sites and corresponding maximum depositional ages: 1—this work; 2—[10]; 3—[11]; 4—[12,13]; 5—[14]; 6—[15]. (**C**) Schematic geological map of the southern part of the Kryvyi Rih Basin.



**Figure 2.** Schematic stratigraphy of the Middle Dnieper Domain of the Ukrainian Shield (after [9], with authors' corrections). The wavy lines indicate unconformities. The ages of the stratigraphic units are given according to [5,10–13,19–21].

The stratigraphic relationships between volcanogenic-sedimentary rocks of the Kryvyi Rih Group and the successions comprising greenstone structures in the Middle Dnieper Domain of the Ukrainian Shield have been discussed by many researchers [16,22–25]. Although there are some apparent differences between the Kryvyi Rih-Kremenchuk Basin and greenstone belts in the Middle Dnieper Domain, which are primarily manifested in the geological sections and the size of the BIF component, some authors have emphasized the certain similarities between the basin and greenstone belts to the extent that the Basin was described as an "atypical greenstone belt" [4,16]. In this regard, the Lykhmanivka and Zhovta Richka tail-shaped structures (Figure 1), adjacent to the Kryvyi Rih-Kremenchuk Basin and linked, respectively, to the Vysokopillya and Verkhivtseve greenstone belts to the east, are of particular interest. The lower parts of their sections comprise volcanogenic and sedimentary rocks of the Konka Group, whereas the upper parts seem to belong to the Kryvyi Rih Group. Hence, parts of the typical sections of the greenstone belts and the Kryvyi Rih-Kremenchuk Basin are combined in the sections of these tail-shaped structures, allowing investigation of their spatial and temporal relationships and construction of the model of sedimentation in the Middle Dnieper Domain in the Mesoarchean and Neoarchean.

In this paper, we report the results of the first geochronological (U-Pb and Hf isotopes) study of detrital zircons separated from two samples of metaterrigenous rocks of the Lykhmanivka Syncline, which extends from the Main Syncline of the Kryvyi Rih Basin towards the Vysokopillya Greenstone Structure (Figure 1). We intended to confirm the temporal relationships between the two main stages of sedimentation in the Middle Dnieper Domain of the Ukrainian Shield and to establish the provenance of the detrital material. In order to achieve these goals, we performed U-Pb dating and Hf isotope measurements in detrital zircon. These data allow us to establish the maximum depositional age of the sediments and their possible sources and compare these results with previously obtained

data for the Kryvyi Rih-Kremenchuk Basin and greenstone belts in the Middle Dnieper Domain of the Ukrainian Shield.

#### 2. Geological Structure of the Studied Area

The Lykhmanivka Syncline is located in the southernmost part of the Kryvyi Rih– Kremenchuk Basin [25,26]. It is a narrow and strongly compressed syncline composed of rocks of the Kryvyi Rih Group, which extends for 30 km from the city of Kryvyi Rih (station of Mykolo-Kozelsk) to the village of Vysokopillya. The width of the syncline varies from 0.5 km in the north to 2 km in the south. In its northern part, the Lykhmanivka Syncline extends into the Kryvyi Rih Main Syncline, and to the south it reaches the Vysokopillya Greenstone Structure. The base of its section is composed of an up to 40 m-thick medium- to coarse-grained amphibolite horizon. Above, a 40–150 m-thick bed of arkoses and phyllites of the Skelevate Formation occurs, overlain by a talc horizon. The Saksahan Formation within the syncline is 50–300 m thick and comprises two horizons: (1) ferruginous hornfels and slates, 100–150 m thick; and (2) jaspillite. Chlorite and carbonaceous slates as well as dolomites of the Hdantsivka Formation overlie the jaspillite horizon, composing a layer up to 200 m thick that comprises the core of the syncline.

The Vysokopillya Greenstone Structure is a monocline characterized by steep rock bedding and tectonic contacts between the different parts of the stratigraphic section [25]. The northern part of the structure is composed of a thick and relatively homogeneous horizon of metamorphosed tholeiitic basalts (Sura Formation, lower sub-formation). Its thickness varies from 2.5 km in the west to 1 km in the east. Fine-grained amphibolites predominate, while amygdaloidal amphibolites are less common. The horizon is complicated by tectonic disturbances, intrusions of ultramafic rocks, and rhyolite and dacite dykes. An older gneiss complex occurs beneath the amphibolite horizon. The metamorphosed volcanic rocks of the Chortomlyk Formation occur higher in the section, forming a layer up to 500 m thick. It is composed of metaandesite with subordinate interlayers of rhyodacites and tholeiitic basalts. The subvolcanic felsic rhyolite-dacite association (Solone Formation) comprises veins and dykes cutting through the tholeiitic basalt. The metarhyodacite dykes reach a thickness of up to 100 m. To the south along the strike, parts of the Vysokopillya Structure are composed of a schist and gneiss association with isolated thick layers of ferruginous-siliceous rocks. The association comprises a heterogeneous sequence of paraand orthogneisses of primary sedimentary (greywacke, subgreywacke, and melanowacke) and volcanogenic (tuff-sandstone, lava breccias, dacite, and andesite with subordinate tholeiite interlayers) origin. In addition to ferruginous-siliceous rocks, which compose thin interlayers, there is a thick steeply dipping metagreywacke bed with an apparent thickness of up to 100 m, composed of a quartz-magnetite-cummingtonite schist with garnet; in places, the garnet content reaches 30–40%. The schists are relatively poor in iron as for BIF, with a total iron oxide content reaching up to 30–32% and a magnetite iron content of 15–18%. In the section, the garnet-bearing quartz-magnetite-cummingtonite schists alternate with metasandstones.

#### 3. Studied Samples

This study is based on U-Pb LA-ICP-MS dating and Hf isotope measurements in detrital zircon isolated from two metasandstone samples. The studied samples were collected in the northern part of the Lykhmanivka Syncline (Mykolo-Kozelsk area, Sample 87-551) where it joins with the Kryvyi Rih–Kremenchuk Basin, and in the central along-strike part of the syncline (Vysokopillya area, Sample 87-222), where the metasediments belonging to the Kryvyi Rih Group rest upon the metavolcanic rocks of the Konka Group (Figure 3). Samples were collected from approximately the same stratigraphic level of the Skelevate Formation of the Kryvyi Rih Group in order to define their maximum depositional ages and to trace possible variations in their provenance.



Figure 3. Schematic logs of drillholes 20803 (Mykolo-Kozelsk area, [27]) and 20586 (Vysokopillya area, [28]).

Considering the moderate degree of metamorphism that inevitably caused some Pb loss and affected U-Pb ages, we did not intend to provide a robust analysis of the detrital age spectrum. Such an analysis is to a large degree meaningless in the metamorphosed detrital sediments, especially taking into account the possibility of the presence of newly formed metamorphic zircon populations. Hence, we did not try to detect all small age groups, because their interpretation will never be certain. Our study was limited by a relatively small amount of analyzed zircon grains (close to 50), to define the major zircon age groups. In this study, we omitted all zircons that yielded more than 10% discordant ages. In most cases, the degree of discordance was less than 5%.

### 4. Research Methods

Zircon was separated using a shaking table, heavy liquids, and a magnetic separator to produce a heavy non-magnetic fraction. Zircons were hand-picked under a binocular microscope and their morphology was studied under an optical microscope. The U-Th-Pb analyses using laser ablation-inductively coupled mass spectrometry (LA-ICP-MS) on zircon crystals in epoxy mounts were performed at the Department of Geology, Trinity College, Dublin, Ireland. A Photon Machines Analyte Excite 193 nm ArF excimer laserablation system with a HelEx 2-volume ablation cell (Teledyne Photon Machines, Belgrade, MT, USA), coupled to an Agilent 7900 mass spectrometer (Agilent, Santa Clara, CA, USA), was used. Line scans on NIST612 standard glass were used to tune the instrument, by obtaining a Th/U ratio close to unity and low oxide production rates (i.e., ThO<sup>+</sup>/Th<sup>+</sup> typically <0.15%). A 24 µm circular laser spot, a 11 Hz repetition rate, and a 2.25 J/cm<sup>2</sup> fluence were used. The helium carrier gas was fed into the laser cell at ~0.4 l/min and was mixed with ~0.6 l/min Ar make-up gas and 11 mL/min N<sub>2</sub>. Each analysis comprised 27.3 s of ablation (300 shots) and 12 s of washout time and the latter portions of the washout were used for baseline measurements. The data reduction of raw U-Th-Pb isotopic data was performed using the freeware package IOLITE [29], with the "Vizual Age" data reduction scheme [30]. The primary U-Pb zircon calibration reference material was 91500 zircon ( $^{206}$ Pb- $^{238}$ U age of 1065.4  $\pm$  0.6 Ma, [31,32] and the secondary reference materials were Plešovice zircon ( $^{206}$ Pb- $^{238}$ U age of 337.13  $\pm$  0.37 Ma, [33]) which yielded an age of  $338.7 \pm 1.0$  Ma (<sup>206</sup>Pb-<sup>238</sup>U age weighted mean age, n = 109) and WRS 1348 zircon  $(^{206}\text{Pb}-^{238}\text{U} \text{ age of } 526.26 \pm 0.70, [34])$  which yielded an age of  $526.6 \pm 2.0$  Ma  $(^{206}\text{Pb}-^{238}\text{U}$ age weighted mean age, n = 130). Final ages were calculated using Isoplot [35].

Hafnium isotope analyses in concordant zircon grains were performed by LA-MC-ICP-MS at the MILESTONE Laboratory (RéGEF ISOTOP-MTP, Geosciences Montpellier, Montpellier, France). A Thermo Scientific Neptune XT (ThermoFisher Scientific, Waltham, MA, USA) was coupled to a Teledyne Cetac Analyte Excite+ Excimer laser (193 nm, Teledyne Photon Machines, Belgrade, MT, USA), which was equipped with an optional X-Y Theta dynamic aperture allowing rectangular-shaped beams of any aspect ratio and orientation to be generated. Analyses were carried out on top of the U-Pb ablation pits, using a 40  $\times$  40  $\mu$ m beam, a laser frequency of 5 Hz, and an energy density of 6 J/cm<sup>2</sup>. Each analysis included a 30 s background measurement and a 60 s ablation period of 60 cycles of 1 s each. The accuracy and long-term reproducibility of the measurements were determined by performing repeated analyses of three zircon reference standards: Mud Tank  $(^{176}\text{Hf}/^{177}\text{Hf} = 0.282512 \pm 17, n = 55)$ ; Plešovice  $(^{176}\text{Hf}/^{177}\text{Hf} = 0.282485 \pm 15, n = 57)$ ; and Temora-2 ( ${}^{176}$ Hf/ ${}^{177}$ Hf = 0.282673 ± 24, *n* = 29). The data agree with the accepted  $^{176}$ Hf/ $^{177}$ Hf ratios for Mud Tank (0.282504 ± 44, [35]), Plešovice (0.282482 ± 13, [33]), and Temora-2 (0.282680  $\pm$  24, [36,37]). All errors are given at the 2 $\sigma$  level. <sup>176</sup>Hf/<sup>177</sup>Hf initial ratios were calculated using the  $^{176}$ Lu decay constant quoted in [38].  $\epsilon$ Hf<sub>(t)</sub> values were calculated using  ${}^{176}Lu/{}^{177}Hf = 0.0336$  and  ${}^{176}Hf/{}^{177}Hf = 0.282785$  for the CHUR [39].

# 5. Results

Zircon from the Skelevate Formation metasandstone (drillhole 20803, int. 88–101 m, Sample 87-551) (Figure 3) is represented by variably rounded, light pink and light brown, transparent and translucent crystals that range in size from  $0.10 \times 0.15$  mm to  $0.25 \times 0.4$  mm (Supplementary Figure S1).

A total of 45 detrital zircon crystals were dated. Twenty of them were over 10% discordant and were omitted from further discussion. The dated zircon grains can be divided into four age groups: a group of 10 crystals had a concordia age of  $3201 \pm 12$  Ma; a group of 6 crystals had a concordia age of  $3089 \pm 11$  Ma; 6 crystals yielded a concordia age of  $2939 \pm 8$  Ma; and the concordia age of the remaining two crystals was  $2059 \pm 4$  Ma (Supplementary Table S1, Figure 4).

Zircons belonging to different Archean age groups have variable initial Hf isotope compositions (Figure 5, Supplementary Table S2). The <sup>176</sup>Hf/<sup>177</sup>Hf ratio gradually increases with decreasing age: in zircons from the oldest ( $3201 \pm 12$  Ma) group, the <sup>176</sup>Hf/<sup>177</sup>Hf ratio varies from 0.280792 to 0.280728 ( $\epsilon$ Hf = 2.8 to 0.2) (Figure 5); in the second ( $3089 \pm 11$  Ma) group, it varies from 0.280812 to 0.280745 ( $\epsilon$ Hf = 1.0 to -1.3); and in the third Archean group ( $2939 \pm 8$  Ma), it ranges from 0.280853 to 0.280848 ( $\epsilon$ Hf = -1.3 to -1.4). Two Paleoproterozoic



zircons yielded very different initial  $^{176}$ Hf/ $^{177}$ Hf ratios: 0.281193 and 0.280774 ( $\epsilon$ Hf = -9.6 and -23.3, respectively).

**Figure 4.** U-Pb concordia diagram and age distribution diagrams plotted as probability density curves for detrital zircon from the metasandstone samples 87-551 and 87-222.



**Figure 5.** Hafnium isotope composition of zircon from two metasandstone samples. Panel (**A**) demonstrates variations in <sup>176</sup>Hf/<sup>177</sup>Hf<sub>(t)</sub> plotted against <sup>207</sup>Pb/<sup>206</sup>Pb in individual zircon grains, whereas panel (**B**) shows variations in  $\varepsilon$ Hf values calculated against the U-Pb ages of each zircon group (see Figure 4). The regression line with <sup>176</sup>Lu/<sup>177</sup>Hf = 0 reflects the evolution of hafnium isotopic composition due to a simple loss of radiogenic lead (zircons lying on this line have the same hafnium isotopic composition regardless of age). The regression line with <sup>176</sup>Lu/<sup>177</sup>Hf = 0.02 reflects the evolution of the hafnium isotopic composition in the 3.56 Ga old mafic source.

The Skelevate Formation at the southern end of the Lykhmanivka Syncline overlies volcanogenic rocks of the Vysokopillya Greenstone Structure. Detrital zircon from a Skelevate Formation metasandstone in this region (drillhole 20586, depth 154–164 m, Sample 87-222) is represented by variably rounded, light pink and light brown, transparent and translucent to opaque crystals of up to  $0.25 \times 0.4$  mm in size. A total of 50 zircon grains were dated; 5 of them yielded more than 10% discordant ages and were omitted from further considerations. In total, 40 results were plotted at the regression line that intercepts the concordia curve at  $3174 \pm 13$  Ma (Figure 4). One grain has a concordant age of  $3583 \pm 14$  Ma, and four others are of Paleoproterozoic age, with the upper intercept at 2038  $\pm 9$  Ma (Supplementary Table S1, Figure 4).

All zircons belonging to the 3174  $\pm$  13 Ma group have similar Hf isotope compositions: the initial <sup>176</sup>Hf/<sup>177</sup>Hf ratio varies from 0.280783 to 0.280754 ( $\epsilon$ Hf = -1.2 to 1.6). Paleoproterozoic zircons have a uniform initial <sup>176</sup>Hf/<sup>177</sup>Hf ratio of 0.281145–0.281131 ( $\epsilon$ Hf = -12.2 to -11.7). The only Paleoarchean zircon has a slightly negative  $\epsilon$ Hf value of -1.5 (initial <sup>176</sup>Hf/<sup>177</sup>Hf = 0.280420, Supplementary Table S2, Figure 5).

## 6. Discussion

#### 6.1. Sources of the Archean Detrital Zircons

The U-Pb dating results demonstrate that the source area was dominated by the gneisses of the Auly Group and granitoids of the Dnipro Complex (3.27–3.18 Ga; [20,21]) and the first intrusive phase of the Sura Complex (3.17–3.1 Ga; [40–42]) (60% and 30%, respectively); zircon from plagioclase granites of the second intrusive phase of the Sura Complex (3.09–2.94 Ga) only accounted for up to 5% of grains.

The hafnium isotope composition (Figure 5, Supplementary Table S2) of zircons from Sample 87-551 suggests that all three Archean groups probably originated from similar rock types that crystallized at different times from the same mafic source (lower crust) with a <sup>176</sup>Lu/<sup>177</sup>Hf ratio of about 0.020 [43]. The results of the hafnium isotopic composition determinations of zircon from Sample 87-222 demonstrate that the spread of U-Pb ages in the predominant age group (3174  $\pm$  13 Ma) along the regression line is due to the partial loss of radiogenic lead. All zircons in the group have nearly identical initial <sup>176</sup>Hf/<sup>177</sup>Hf ratios which do not vary with age. In terms of U-Pb ages and Hf isotope compositions, the detrital zircons from the two oldest age groups in Sample 87-551 and the predominant (old) age group in Sample 87-222 are very similar.

### 6.2. The Maximum Depositional Age and Origin of the Paleoproterozoic Zircon

The amount of Paleoproterozoic zircons dated at 2.07–2.03 Ga in the studied samples is only ca. 5%. However, their origin raises an important question as the maximum depositional age of the studied rocks depends on the nature of these zircons. If they are detrital, then the maximum depositional age of the entire Kryvyi Rih Structure would be Paleoproterozoic. However, if they were formed in response to Paleoproterozoic metamorphism, then the maximum depositional age of the studied rocks must be ca. 2.9 Ga.

The Paleoproterozoic zircons in the two metasandstone samples differ from their Archean counterparts by a low Th/U ratio indicating their metamorphic origin (Figure 6; [44–46]). In contrast to the Archean zircons, the Paleoproterozoic ones are enriched in U and Th (Supplementary Table S1). In terms of their Hf isotope composition, most of the Paleoproterozoic zircons have rather homogeneous  $\varepsilon$ Hf values (–12.2 to –9.6; average –11.4 ± 1.8) which indicates the input of crustal fluids with an isotope composition that has evolved since Archean times with a <sup>176</sup>Lu/<sup>177</sup>Hf ratio close to 0.02.

Most of the studied samples of terrigenous rocks of the Kryvyi Rih Group contain no Paleoproterozoic zircons ([10,13]. However, Paleoproterozoic zircons were previously identified in metasedimentary rocks of the Hleyuvatka Formation [19], and in amphibolite (metabasalt) of the Novokryvorizka Formation [11]. The zircons with a metamorphic protolith in the metasedimentary rocks of the Hleyuvatka Formation were considered as detrital, while those from amphibolite were considered as metamorphic. Unfortunately, there is no way to distinguish detrital zircons derived from a metamorphic rock from metamorphic zircons crystallized within a metasedimentary rock [47,48]. Considering this, we rely on indirect arguments to define the origin of the Paleoproterozoic zircons with a metamorphic origin found in our metasandstone samples.



**Figure 6.** Variations in the Th/U ratio between zircons of different ages from the two metasandstone samples.

First, we consider the in situ metamorphic growth of Paleoproterozoic zircons in the metasandstone samples. Paleoproterozoic metamorphic zircons are common in strongly metamorphosed Archean rocks of the Middle Bouh Domain of the Ukrainian Shield [49,50]. In these rocks, Paleoproterozoic zircon forms overgrowths on older cores or occurs as newly formed crystals. Some zircon crystals that yielded Paleoproterozoic ages appear to have the same hafnium isotopic composition as Archean zircons, indicating that the apparent Paleoproterozoic age of these crystals resulted from the loss of radiogenic lead. However, the hafnium isotope composition in most of the Paleoproterozoic metamorphic zircons is more radiogenic, suggesting that either juvenile fluids enriched in radiogenic hafnium were involved in their crystallization, or radiogenic hafnium was, due to metamorphic reactions, derived from other minerals with a high Lu/Hf ratio [51]. A similar situation is observed in our metasandstone samples from the Skelevate Formation—their hafnium isotopic composition is significantly more radiogenic than would be expected in the case of radiogenic lead loss.

The second possibility is that Paleoproterozoic zircons are detrital grains. In general, zircons from Paleoproterozoic granitoids and metamorphic rocks of the Ukrainian Shield are characterized by a juvenile hafnium isotopic composition [52,53], although some Paleoproterozoic granitoids with a long crustal residence history are also known [54]. Considering the widespread occurrence of Paleoproterozoic granites and metamorphic rocks [55–57], and the wide variability in their Hf isotopic characteristics with a predominance of juvenile Hf isotopic values, we would expect to find many more Paleoproterozoic detrital zircons than those that have actually been found and with wider variations in hafnium isotopic composition. Hence, we assume that the Paleoproterozoic zircons in the studied detrital rocks must be in situ metamorphic/metasomatic in origin, rather than detrital. Thus, the maximum depositional age can be determined at 2.9 Ga.

#### 6.3. Variations in the Provenance of the Sediments in the Kryvyi Rih–Kremenchuk Basin

An important feature of the studied rocks is a variation in the sources of detrital material. Despite the relative spatial proximity of the two sampling sites and their identical stratigraphic position, they reveal a different provenance. Moreover, they differ from other metasedimentary rocks of the Skelevate Formation studied previously [13]. These variations indicate the local nature of sedimentation and the absence of significant lateral transport and mixing of detrital material within the sedimentary basin.

Based on detrital zircon studies from rocks of the Kryvyi Rih and Inhul-Inhulets Groups from the Kryvyi Rih–Kremenchuk Basin (Zhovte region) in the north (Figure 1) to the Vysokopillya Greenstone Structure in the south, important regularities in the distribution of the source rocks have been established. Detrital zircon dated at 2.68 Ga was documented in sandstones of the Inhul-Inhulets Group (a possible stratigraphic equivalent of the Kryvyi Rih Group) in the Pravoberezhna region [15]. Their source remains unknown, and the nearest possible provenance is represented by metarhyolites of the Lebedinska Group (2.62 Ga) and granitoids of the Atamanskyi Complex (2.6–2.4 Ga) that occur in the Voronizh Crystalline Massif. In the East Hannivka Belt and the Saksahan Syncline, the composition of metasandstones of the Skelevate Formation is characterized by significant zircon derived from the Saksahan plagioclase granites (3.06 Ga) [13,14]. Sediments of the Lykhmanivka Syncline are dominated by detrital zircon derived from rocks of the Sura Complex (3.17–3.10 Ga). Detrital zircon with distinct peaks at 3.07, 3.19, 3.26, and 3.32 Ga has been found in metasandstones overlying the Vysokopillya Greenstone Structure [58].

#### 6.4. Relationships between the Kryvyi Rih-Kremenchuk Basin and Greenstone Belts

Greenstone belts occur widely in the Precambrian continental crust worldwide and represent an important stage in the evolution of the crust, although their origin and tectonic setting are not fully understood [59–61]. The Middle Dnieper Domain of the Ukrainian Shield represents a typical granite–greenstone terrane that hosts several greenstone belts [4]. Although each belt demonstrates some peculiarities of structure and composition, they all have similar patterns in their sections. The bottom parts of the belts are dominated by mafic and ultramafic volcanic rocks which are overlaid by intermediate to felsic volcanic rocks and then by siliciclastic metasediments and banded iron formation. The available geochronological data [5,40,42,58,62–64] indicates that the formation of the greenstone belts has finished by ca. 2.9 Ga.

The time interval between 2.9 Ga and 2.7 Ga is known in the Ukrainian Shield as a period of metamorphism and emplacement of numerous granitic intrusions [40,50,63,65–68]. In the Middle Dnieper Domain, this stage is characterized by the emplacement of large granite massifs belonging to the Tik, Demuryne, and Saksahan Complexes. Obviously, granite emplacement was accompanied by deformation and metamorphism of the previously formed rock complexes.

Although some researchers [4,16] tend to parallelize the Kryvyi Rih–Kremenchuk Basin with greenstone belts, it is apparent that their formation was separated by a significant period of time, as the maximum deposition age of the siliciclastic sediments in the basin does not exceed ca. 2.8 Ga. The age of the sedimentation of the Kryvyi Rih–Kremenchuk deposits is poorly defined as Neoarchean to Paleoproterozoic (e.g., [69]). Our data confirm that the sedimentary basins superimposed on the greenstone belts were filled in with the material derived from local sources, including the felsic volcanic and subvolcanic rocks associated with the greenstone structures.

# 7. Conclusions

Detrital zircon U-Pb dating results from metaterrigenous rocks of the Skelevate Formation in the Lykhmanivka Syncline indicate that metasandstones in the northern part of the syncline contain zircons belonging to four age groups:  $3201 \pm 12$  Ma,  $3089 \pm 11$  Ma,  $2939 \pm 8$  Ma, and  $2059 \pm 4$  Ma. In contrast, zircon from metasediments from the southern end of the Lykhmanivka Syncline fall into two age groups:  $3174 \pm 13$  Ma, and  $2038 \pm 9$  Ma. The main sources of the detrital material comprise metamorphic rocks of the Auly Group, granites of the Dnipro Complex, and the first intrusive phase of the Sura Complex.

The amount of Paleoproterozoic zircons in the studied rocks does not exceed 5% and they are considered to be in situ metamorphic/metasomatic in origin. This allows us to define the maximum depositional age of the metaterrigenous rocks of the Lykhmanivka Syncline at ca. 2.9 Ga. These data are in good agreement with the previously obtained results for metaterrigenous rocks of the Kryvyi Rih–Kremenchuk Basin. Our findings indicate local sedimentation with minimal lateral transport and mixing of detrital material within the sedimentary basin.

**Supplementary Materials:** The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/geosciences14100254/s1, Supplementary Table S1: Detrital zircon U-Pb data from metasandstones of the Lykhmanivka structure of the Middle Dnieper Domain; Supplementary Table S2: Detrital zircon Hf isotopic data from metasandstones of the Lykhmanivka structure of the Middle Dnieper Domain. Supplementary Figure S1: Optical images of the studied zircon crystals from metasandstone samples 87-551 and 87-222 with the position of the U-Pb analytical spots indicated.

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**Data Availability Statement:** All the analytical data are presented in the paper, including the Supplementary Materials.

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