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Communication

The Meteoritic Origin of Morocco Iron Dagger Blades

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Abstract: Up until now, a few artifacts made of meteoritic iron have been discovered worldwide, though none in Morocco. The number of these objects has rarely been verified, as museums generally do not allow artifacts to be tested, and they are often confused with common smelted objects of the Iron Age. In this work, portable X-ray fluorescence (pXRF) and scanning electron microscopy-energy dispersive X-ray spectroscopy (SEM-EDS) have been used to analyze three iron dagger blades recovered in two localities near Imilchil and Missour in Morocco. The composition of one blade (7.2 wt% Ni and 1.1 wt% Co) strongly supports its meteoritic origin, whereas it was not so for the other two ones. The results of this work provide the first case of the exploitation of meteoritic iron as a metal source in Morocco.

Keywords: iron artifacts; meteoritic iron; portable X-ray fluorescence; scanning electron microscopy-energy dispersive X-ray spectroscopy

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1. Introduction

A long unresolved and controversial debate exists concerning the meteoritic (extraterrestrial) or smelted (terrestrial) origin of Bronze Age iron artifacts [1]. Although the Iron Age began in Anatolia and the Caucasus around 1200 BCE, nearly 2000 years earlier various cultures were already using iron objects. These items were extremely rare and were always considered very precious, although iron ores abound on the terrestrial surface.

Only a few detailed scientific studies have reported the identification of meteoritic iron in ancient artifacts convincingly. Among these are two funerary iron bracelets and an ax excavated from two different Polish archaeological sites [2] and an ancient "iron man" Buddhist sculpture carved from a fragment of the Chinga meteorite [3]. Probably the most important discovery was that of Comelli et al. [4] regarding the meteoritic origin of the iron dagger blade recovered in the sarcophagus of the ancient Egyptian King Tutankhamen (14th C. BCE), which was the subject of many debates due to previous analyses yielding controversial results. The study [4] determined accurately, using portable X-ray fluorescence (pXRF), the composition of the blade (Fe plus 10.8 wt% Ni and 0.58 wt% Co), which strongly supports its meteoritic origin and confirms that ancient Egyptians attributed a great value to meteoritic iron for the production of precious objects.

Besides gathering already available data, Jambon [1] used a pXRF spectrometer to reveal that several Bronze Age iron artifacts in his collection were made with meteoric iron. These included beads from Gerzeh (Egypt, −3200 BCE), a dagger from Alaca Höyük (Turkey, −2500 BCE), a pendant from Umm el-Marra (Syria, −2300 BCE), an ax from Ugarit (Syria, −1400 BCE), and several others from the Shang dynasty civilization (China, −1400 BCE), and the dagger, bracelet, and headrest of Tutankhamen (Egypt, −1350 BCE).

When large celestial bodies such as the Earth formed, nearly all Ni drifted towards the molten iron core; thus, it is extremely rare to find Ni on the surface. In contrast, meteorites originating from celestial bodies shattering can be composed of a core material containing Fe and high levels of Ni and Co, which makes it possible to identify the source of iron [1]. More recently, Chen et al. [5] confirmed the meteoritic origin of two Bronze Age bimetallic iron blades from central China, shedding new light on the perception of hot-work processing of ancient meteoritic iron items.

In Morocco, no artifacts made of meteoritic iron have been found up to now. However, recent studies by Ibhi et al. [6], Nachit et al. [7], and Moggi-Cecchi et al. [8] have shown the existence of several iron meteoritic fields in the North and North West of Morocco, mostly in the Agoudal (Imilchil) and Maatarka (Taza) areas. Thousands of small pieces (1–200 g) and many 200–1000 g fragments (with the largest piece of 196 kg) have been recovered in the Agoudal area by systematic searching with metal detectors. The majority of Agoudal meteorite fragments were found in a strewn field roughly 6 × 2 km in extent [6]. The total recovered mass of Oglat Sidi Ali meteorite fragments recovered in the Maatarka region is estimated to amount to more than 1500 kg, including a 190 kg piece, spread across the NE–SW oriented 20-km long and 5-km large strewnfield [8].

Out of several dozen artifacts recovered by a French antique dealer during an expedition for meteorite and antique objects recovery in Morocco, only four artifacts responded positively to the dimethylglyoxime (DMG) test for the presence of Ni. However, this test leads to "false positives"; thus, it is not adequate for the unambiguous identification of meteoritic origin, as many industrial iron objects may contain enough Ni to generate a positive response to the DMG test.

In the present paper, three iron dagger blades found in 2020 near Imilchil and Missour (a city near Maatarka) were analyzed by pXRF and scanning electron microscopy energy-dispersive X-ray spectroscopy (SEM-EDS) with the aim of (i) identifying the nature of artifacts by measuring their textural features, mineralogy and main metal composition, and (ii) discussing the possible genetic relationship with iron meteorites collected in the corresponding regions.

2. Materials and Methods

The sample objects of this work were three iron dagger blades recently (2020) found in localities near Imilchil and Missour in Morocco. In particular, the three artifacts, A (dimensions, 23.3 cm × 2.5 cm × 0.3 cm; weight, 98.5 g), B (dimensions, 14 cm × 1.5 cm × 0.2 cm; weight, 27.6 g) and C (dimensions, 8.5 cm × 1–1.3 cm × 0.2 cm; weight, 15.1 g), were very altered (Figure 1) by weathering effects due to their environment and age. All measurements were performed on polished sections of the blades. Two classified iron meteorites found in the same areas were used for comparative purposes, i.e., an iron meteorite named Agoudal, discovered nearby the village of Imilchil in the High Atlas Mountains and classified in the IIAB magmatic iron group [9,10], and the Oglat Sidi Ali iron meteorite that was recovered in 2013 near the locality of Maatarka, Morocco [8,11].

The structural, mineralogical, and compositional features of the three artifacts were determined by scanning electron microscopy—energy-dispersive X-ray spectroscopy (SEM-EDS) using a JEOL JSM IT1000 at the Scientific Research Center of the Faculty of Sciences of the Ibn Zohr University. The analyses were performed using a magnification of 4 nm at an accelerating voltage of 20 kV. The Ni and Co compositions of the three iron dagger blades and the two meteorites were confirmed by measurements with a pXRF instrument, i.e., a Niton XL3t 950 analyzer (Thermo-Fisher Scientific, Waltham, MA, USA, Table S1). The measurement conditions used in all cases were: working distance, ~1 cm; tube voltage, 50 kV; tube anode current, 80 µA; and acquisition time, 60 s. Repeated spectral measurements were taken at two different spots of about 3-mm-diameter on the dagger blades.

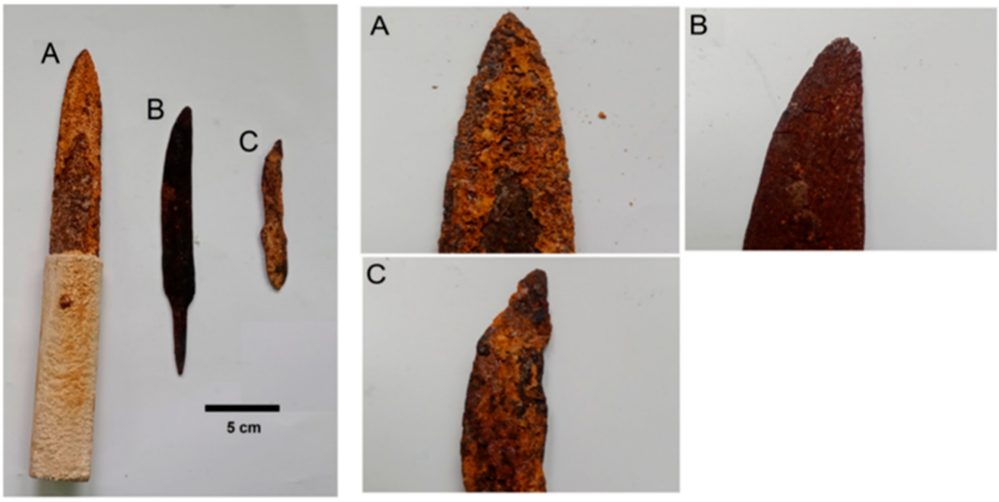

Figure 1. The three blade daggers analyzed in this study. **Left**: full-size images. **Right**: detailed images exhibiting strongly oxidized rusty and weathered surfaces.

3. Results and Discussion

All three blade samples show a marked magnetism and feature severely corroded conditions, with numerous particles of remnant metal displaying twists and kneaded belts in a matrix of weathering products (Figure 1). The entire surface of knife A appeared very weathered and covered with layers of a rusty deposit, which indicated that it had been subjected to extended alterations, most likely by humidity and soil, for years. SEM-EDS analysis of the clean surface showed that it is composed of hydrated Fe oxides with an average Ni content of 0.95 wt%. The elements present at levels lower than 1 wt%, such as Ca, Mn, and Cu, are likely to derive from soil contamination. No Co was detected. Knife C was the smallest one with a particular shape, i.e., both the handle and the blade are made of metal with a curved end, in which neither Ni nor Co were detected. Conversely, the EDS analysis of knife B revealed: 7.2 wt% of Ni and 1.1 wt% of Co. SEM analysis of a plate (~2 g) cut from the scaled tip of blade B (Figure 2) showed an intergrowth of kamacite and taenite. These lamellae consisted of multiple kamacite (Ni, 5.2 wt%) spindles roughly parallel to each other and ranging in width from 20 to 100 μm, separated by thin, Ni-rich (Ni 34.5 wt%) taenite.

The Ni content in most iron meteorites ranges from 5 wt% to 35 wt%, whereas it never exceeds 4 wt% in artificial iron artifacts [12]. Most weathered meteorite materials are composed of Ni and ferrous Fe oxides and oxyhydroxides γ-(Fe, Ni)$_2$O$_3$ [13] and form via Ni substitution for Fe, where little loss of Ni occurs during magnetite formation. Ni is preferentially leached during weathering, whereas Fe oxidized to Fe^{3+} is not. The Ni/Co ratio remains constant for mild weathering and then decreases when part of the Co is oxidized to Co^{3+} [1].

No other minerals, such as graphite, troilite, or schreibersite, were detected (Figure S1). The texture was that of a plessitic octahedrite with some distortion because of the cold working of this material (Figure 3a).

The pXRF measurements carried on the surface of the dagger blade B showed that Fe and Ni are its main bulk constituents, whereas the other blades contained only <0.95 wt% Ni, so they were not of meteoritic origin.

Figure 2. The blade dagger B analyzed in this study. (**Top**): full-size image. (**Bottom**): detailed images that exhibit the strongly oxidized rusty and weathered surface and the plate removed from the scaled tip of the blade.

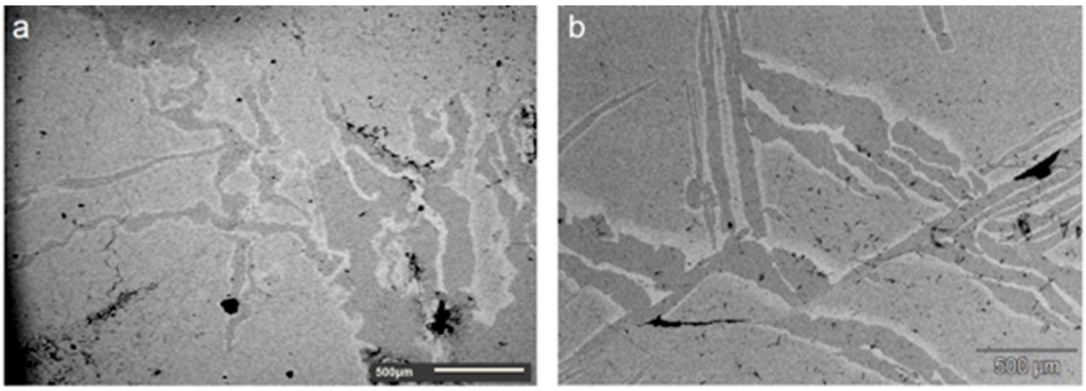

Figure 3. SEM images of (**a**) an area of the polished sample tip of dagger blade B and (**b**) of Oglat Sidi Ali meteorite [8], where dark grey is kamacite and pale grey is taenite.

Genetic Relationship with Meteorites Collected in the Region

The possible genetic relationship of blade B with iron meteorites was evaluated by comparing it with two meteorite samples collected in Agoudal and Oglat Sidi Ali meteorite fields located, respectively, 50 and 100 km from the place where the daggers were recovered. The SEM analyses of the Oglat Sidi Ali meteorite showed a very fine-scale pattern of lamellae consisting of multiple tiny kamacite spindles roughly parallel to each other and ranging in width from 30 to 80 µm, which were separated by thin, Ni-rich taenitic spindles that together formed a plessitic octahedrite arrangement (Figure 3b). No other minerals, such as graphite, troilite, or schreibersite, were observed. SEM-EDS analyses yielded a mean Ni content of 147 $\pm$ 0.2 mg/g in taenite, with a maximum of 452 $\pm$ 0.3 mg/g at spindle borders, while the Ni-poor iron phase, i.e., kamacite, had a mean Ni content of 67 $\pm$ 0.1 mg/g [8]. More recent data obtained for the Oglat Sidi Ali meteorite by inductively coupled plasma-mass spectrometry (ICP-MS) reported a very similar Ni content, i.e., 14.7 wt%, and a Co content of 1.1 wt% (Table 1) [14]. The SEM-EDS analysis of the Agoudal meteorite revealed a prevalent coarse-grained kamacite and schreibersite structure with average Ni and Co contents of ~5.5 wt% and 0.4 wt%, respectively (Table 1) [9,10].

Table 1. The concentrations of Ni and Co (wt%) in the dagger blade samples and Agoudal and Oglat Sidi Ali iron meteorites.

	Dagger A (±2δ)	Dagger B (±2δ)	Dagger C	Agoudal [a]	Oglat Sidi Ali [b]
Ni	0.95 (±0.019)	7.22 (±0.16)	N.D. [c]	5.5	14.1
Co	N.D. [c]	1.07 (±0.05)	N.D. [c]	0.4	1.1

[a] Data from Ruzicka et al. [9]. [b] Data from Moggi Cecchi et al. [8]. [c] N.D., not determined.

A pairing procedure based on microscopic features and mineralogical and geochemical data was performed by comparing blade B to Oglat Sidi Ali and Agoudal meteorites. The Agoudal meteorite showed no mineralogical and textural relationship with dagger B, whereas the mineralogy and plessitic texture of the Oglat Sidi Ali meteorite was similar to that of blade B, as plates of kamacite are segregated in plessite fields, although the content of Ni was slightly different (Table 1). However, analyzing trace elements, i.e., Ga, Ge, Ru, Pd, and Pt, is necessary for classifying iron meteorites [15] and comparing them with the dagger, so further research is needed to acquire information on the origin of this blade.

4. Summary and Conclusions

The concentrations of the crucial major elements, i.e., Ni and Co, of the two artifacts A and C were in the range of values known for terrestrial iron ores. In contrast, those of artifact B, together with its chemical microstructural composition, were consistent with those of an iron meteorite. In particular, thin fragments of parallel bands of taenite and kamacite with structural distortion were present. Furthermore, the object retained its original composition of approximately 7 wt% Ni and 1 wt% Co, which strongly suggest an extraterrestrial origin. Although the textural and mineralogical data were similar to plessitic octahedrites typical of the Oglat Sidi Ali meteorite, Ni compositional data were slightly different; thus, further research is needed to confirm the origin of this blade.

The discovery of this iron dagger blade in the Moroccan High Atlas would suggest that the ancient exploitation of iron in Morocco involved meteoritic iron as the metal source. This study is the first scientific report on the iron industry in the High Atlas based on fragments of iron meteorites. However, in this region, many other meteoritic artifacts can be found. Therefore cooperation between collectors and the University Museum of Meteorites should be developed to work out methods of researching man-made meteoritic artifacts. According to Kotowiecki [2], meteoritic artifacts should not be neglected because, despite being reworked, they are still of extraterrestrial origin and should be registered as meteorites.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/heritage5030072/s1, Table S1. XRF data for the three dagger blades; Figure S1. SEM-EDS images and data of the two phases analyzed (kamacite and taenite). Other phases (troilite, graphite and schreibersite) are not apparent in the analyzed section.

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Conflicts of Interest: The authors declare no conflict of interest.

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