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The meteoritic origin of Tutankhamun's iron dagger blade

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Abstract–Scholars have long discussed the introduction and spread of iron metallurgy in different civilizations. The sporadic use of iron has been reported in the Eastern Mediterranean area from the late Neolithic period to the Bronze Age. Despite the rare existence of smelted iron, it is generally assumed that early iron objects were produced from meteoritic iron. Nevertheless, the methods of working the metal, its use, and diffusion are contentious issues compromised by lack of detailed analysis. Since its discovery in 1925, the meteoritic origin of the iron dagger blade from the sarcophagus of the ancient Egyptian King Tutankhamun (14th C. BCE) has been the subject of debate and previous analyses yielded controversial results. We show that the composition of the blade (Fe plus 10.8 wt% Ni and 0.58 wt% Co), accurately determined through portable x-ray fluorescence spectrometry, strongly supports its meteoritic origin. In agreement with recent results of metallographic analysis of ancient iron artifacts from Gerzeh, our study confirms that ancient Egyptians attributed great value to meteoritic iron for the production of precious objects. Moreover, the high manufacturing quality of Tutankhamun's dagger blade, in comparison with other simple-shaped meteoritic iron artifacts, suggests a significant mastery of ironworking in Tutankhamun's time.

INTRODUCTION

The working of metal has played such a crucial role in the evolution of human civilization that historians conventionally divide ancient eras into "metal" ages, taking into account the use of copper, bronze, and iron in sequence. However, it is clear that sharp breaks in these periods are conventional. In particular, the start of the iron age has long been discussed.

Ancient Egypt had great mineral resources. The wide desert areas, in particular the Eastern desert, are rich in mines and quarries, which have been exploited since ancient times (Ogden 2000; Klemm and Klemm 2008; Lucas and Harris 2012). Copper, bronze, and gold have been used since the 4th millennium BCE (Ogden 2000). In contrast, despite the significant presence of iron ores in ancient Egypt (Ogden 2000; Lucas and Harris 2012), the utilitarian use of iron in the Nile Valley occurred later than in neighboring countries, with the earliest references to iron smelting dating to the 1st millennium BCE (Tylecote 1992; Waldbaum 1999; Ogden 2000).

The sporadic use of iron during the Bronze Age has been reported in Egypt and the Mediterranean (Photos 1989; Tylecote 1992; Waldbaum 1999; Ogden 2000). A handful of iron objects likely dates to the Old Kingdom (3rd millennium BCE) onward (Waldbaum 1999; Ogden 2000), with the most ancient iron ones dated to about 3200 BCE (Stevenson 2009). It is generally assumed that early iron objects were produced from meteoritic material, despite the rare existence of smelted iron fortuitously obtained as a by-product of copper and bronze smelting (Bjorkman 1973; Photos 1989; Tylecote 1992; Bard 1999; Waldbaum 1999; Ogden 2000). During the Bronze Age, iron was definitely rare, its value was greater than that of gold (Burney 2004), and it was primarily used for the production of ornamental, ritual, and ceremonial objects (Bjorkman 1973; Tylecote 1992; Waldbaum 1999). This suggests that either early iron artifacts were unsuitable for utilitarian and military purposes or working techniques for producing the metal in large quantities had not yet been mastered (Waldbaum 1999). By the end of the 2nd millennium BCE, iron had come into common use in most of the eastern Mediterranean, although the rates at which it was substituted for bronze vary from region to region (Tylecote 1992; Waldbaum 1999; Ogden 2000).

Over the past 50 years, the interest in the use of meteoritic iron and in the introduction and spread of iron smelting technology in the Mediterranean area has increased steadily (Bjorkman 1973; Photos 1989; Tylecote 1992). Different historical and philological studies have addressed these topics (Piaskowski 1982; Photos 1989). Compositional and structural analyses of ancient iron findings have been performed and reported (Bjorkman 1973; Photos 1989; Waldbaum 1999), but despite few cases (Johnson et al. 2013; Rehren et al. 2013), the common lack of detailed information on analytical methods and of robust data hinders their utility in answering broader questions (Photos 1989; Waldbaum 1999). Investigations are further hampered by the difficulty in obtaining permissions to analyze rare and precious artifacts with either destructive or nondestructive techniques (Photos 1989).

Beyond the Mediterranean area, the fall of meteorites was perceived as a divine message in other ancient cultures. It is generally accepted that other civilizations around the world, including the Inuit people; the ancient civilizations in Tibet, Syria and Mesopotamia (Buchwald 2005; Buchner et al. 2012); and the prehistoric Hopewell people living in Eastern North America from 400 BCE to 400 CE, used meteoritic iron for the production of small tools and ceremonial objects (Prufer 1962). Nonetheless, only few detailed scientific analyses have clearly reported the identification of meteoritic iron in ancient artifacts. These include several iron tools made by the Inuit people in Greenland, recognized as being made of small fragments of the Cape York iron meteorite shower (Buchwald 1992); the ancient "iron man" Buddhist sculpture, likely carved from a fragment of the Chinga meteorite (Buchner et al. 2012); two funerary iron bracelets and an axe excavated in two different Polish archaeological sites (Kotowiecki 2004); and, less recently, a few masses of meteoritic iron from the Hopewell culture (Prufer 1962).

Of the rare surviving examples of iron objects from ancient Egyptian culture, the most famous is the dagger from the tomb of the ancient Egyptian King Tutankhamun. The history of King Tutankhamun (18th dynasty, 14th C. BCE) has fascinated scientists and the general public since the discovery of his spectacular tomb in 1922 by archaeologist Howard Carter (Carter and Mace 1923-1927-1933). In 1925, Carter found two daggers in the wrapping of the mummy: one on the right thigh with a blade of iron (Fig. 1) and the other on the abdomen with a blade of gold (Carter and Mace 1923-1927-1933). The former (Carter no. 256K, JE 61585) is the object of our study. The dagger has a finely manufactured blade, made of nonrusted, apparently homogeneous metal (Fig. 2). Its handle is made of fine gold, is decorated with cloisonné and granulation work, and ends with a pommel of rock crystal (Feldman 2006; Zaki 2008). Its gold sheath is decorated with a floral lily motif on one side and with a feathers pattern on the other side, terminating with a jackal's head.[1]

Among the iron objects discovered in Tutankhamun's tomb, which also include 16 miniature iron blades, a miniature head rest, and a bracelet with the Udjat eye of iron,[2] the dagger is the one that has most attracted interest from archaeologists and historians, mainly in relation to the origin of the metal and to the employed working technology (Bjorkman 1973; Photos 1989; Tylecote 1992; Waldbaum 1999; Johnson et al. 2013). As already observed by Carter, the iron objects from Tutankhamun's tomb highlight some innovative features of the use and trade of iron in the Late Bronze Age (Carter and Mace 1923-1927-1933). Interestingly, diplomatic documents from the Egyptian royal archives from the 14th C. BCE (the Amarna letters) mention royal gifts made of iron in the period immediately before the Tutankhamun's reign. In particular, it is reported that Tushratta, King of Mitanni, sent precious iron objects to Amenhotep III,

[1]Carter card: http://www.griffith.ox.ac.uk/gri/carter/256k-c256k-1.html.
[2]Tutankhamun: Anatomy of an Excavation. http://www.griffith.ox.ac.uk/discoveringTut/ (2014).

Fig. 1. The mummy of King Tutankhamun. Black and white picture of Tutankhamun mummy showing the iron dagger (34.2 cm long) placed on the right thigh (arrowed). Copyright Griffith Institute, University of Oxford.

Fig. 2. The iron dagger of King Tutankhamun. Color picture of the iron dagger (Carter no. 256K, JE 61585) with its gold sheath. The full length of the dagger is 34.2 cm.

who may have been the grandfather of Tutankhamun. Daggers with iron blades and a gilded iron hand bracelet are mentioned in the list (McNutt 1990; Morkot 2010; Lucas and Harris 2012; Rainey 2014).

Results of previous analyses of Tutankhamun's iron funerary objects have proved controversial. Bjorkman (1973) referred to a meteoritic origin of the iron dagger on the basis of its high nickel content determined through an analytical study performed in 1970; however, to the best of our knowledge, this study has not been published and the analytical techniques used at that time were not specified. In 1994, analysis of the dagger's iron blade by X-ray fluorescence (XRF) spectrometry revealed a Ni content of 2.8 wt%, which was considered inconsistent with meteoritic iron by the authors (Helmi and Barakat 1995).

Iron meteorites are mostly made of Fe and Ni, with minor quantities of Co, P, S, and C, and trace amounts of other siderophile and chalcophile elements (Haak and McCoy 2003). Their chemical compositions are typically determined by means of sensitive, yet destructive, analytical methods, including instrumental neutron activation analysis (Wasson and Sedwick 1969) and inductively coupled plasma mass spectrometry (D'Orazio and Folco 2003). XRF measurements, carried out in the laboratory and, more recently, with the aid of portable or handheld devices, have been widely used for the bulk

Table 1. Samples analyzed by XRF spectroscopy.

Sample	No. of analysis points	Ni wt%	Co wt%	Estimated Ni wt%	Estimated Co wt%	References
Campo del Cielo	21	6.73	0.46	6.82 ± 0.35	0.52 ± 0.03	Wasson and Kallemeyn (2002)
Canyon Diablo	32	6.93	0.47	7.09 ± 0.34	0.55 ± 0.04	Wasson and Kallemeyn (2002)
Chinga	20	16.5	0.58	15.71 ± 0.33	0.57 ± 0.04	Buchner et al. (2012)
Dronino	12	9.81	0.55	9.83 ± 0.30	0.57 ± 0.04	Russell et al. (2004)
Gebel Kamil	17	20.60	0.76	20.68 ± 0.46	0.70 ± 0.05	D'Orazio et al. (2011)
Gibeon	16	7.99	0.39	7.90 ± 0.33	0.43 ± 0.03	Wasson and Richardson (2001)
Hoba	1	16.30	0.78	17.08 ± 0.36	0.69 ± 0.05	Campbell and Humayun (2005)
North Chile	1	5.65	0.454	6.16 ± 0.36	0.51 ± 0.03	Wasson et al. (1989)
NWA 5289	12	9.02	0.40	9.94 ± 0.30	0.41 ± 0.03	Weisberg (2008)
Tambo Quemado	10	10.15	0.56	9.44 ± 0.31	0.50 ± 0.03	D'Orazio and Folco (2003)
Tres Castillos	7	9.23	0.51	8.57 ± 0.32	0.47 ± 0.03	Wasson et al. (1998)
ARMI AISI 303	1	9.5	0.20	8.65 ± 0.32	0.10 ± 0.03	Reference values from ARMI certificate
NIST SRM 1262b	1	0.60	0.30	1.72 ± 0.48	0.36 ± 0.03	Reference values from NIST certificate
NIST SRM 1158	1	36.10	0.01	40.57 ± 1.15	0.05 ± 0.04	Reference values from NIST certificate
SS-CRM 461/1	4	6.12	n.d.	6.13 ± 0.36	0.04 ± 0.04	BAS Bureau of Analysed Samples Ltd.*
SS-CRM 462/1	4	12.85	n.d.	12.04 ± 0.30	0.04 ± 0.04	BAS Bureau of Analysed Samples Ltd.*
SS-CRM 463/1	4	10.20	0.12	10.20 ± 0.30	0.07 ± 0.04	BAS Bureau of Analysed Samples Ltd.*
SS-CRM 464/1	4	20.05	0.05	20.78 ± 0.46	−0.01 ± 0.04	BAS Bureau of Analysed Samples Ltd.*
SS-CRM 465/1	4	9.24	0.05	9.20 ± 0.31	0.04 ± 0.04	BAS Bureau of Analysed Samples Ltd.*
SS-CRM 466/2	4	10.20	0.02	10.02 ± 0.30	0.01 ± 0.04	BAS Bureau of Analysed Samples Ltd.*
SS-CRM 467/1	4	9.21	n.d.	9.10 ± 0.31	0.02 ± 0.04	BAS Bureau of Analysed Samples Ltd.*
SS-CRM 468/1	4	8.90	0.02	8.89 ± 0.31	0.02 ± 0.04	BAS Bureau of Analysed Samples Ltd.*
Tutankhamun's iron dagger blade	2			10.85 ± 0.30	0.58 ± 0.04	

The list includes 22 reference samples (11 meteorites of well-known composition and 11 certified steel reference materials) and Tutankhamun's iron dagger blade. In the list, we have reported: the number of analysis points; Ni and Co reference concentrations of samples used for XRF calibration; Ni and Co concentrations estimated following linear calibration of XRF data provided within a 95% confidence interval. * http//basrid.co.uk/ (2015)

nondestructive analysis of meteorites since the late 1960s and early 1970s (Reed 1972; Zurfluh et al. 2011; Gemelli et al. 2015).

In this work, we have determined the bulk composition of the Tutankhamun's iron dagger blade using state-of-the-art, nondestructive XRF analysis. In the last 20 years, a dramatic improvement in solid-state detectors technology has allowed new analytical applications. Modern energy dispersive XRF spectrometers exhibit typical energy resolutions below 140 eV @ Mn $K\alpha$ line (West et al. 2013), allowing the deconvolution of close peaks (Redus and Huber 2012), as required for correctly estimating minor amounts of cobalt in meteoritic irons.

MATERIALS AND METHODS

Samples

XRF measurements were performed on Tutankhamun's dagger, 11 meteorites of well-known composition, and 11 certified steel reference materials. The full list of analyzed samples is provided in Table 1. The number of point analyses for each sample is also reported. The location of the two point analyses on Tutankhamun's iron dagger blade is reported in supporting information (Fig. S1).

Portable XRF Spectrometry

The XRF spectrometer (ELIO, XGLab srl, Italy) is based on a 25 mm^2 active area silicon drift detector and on a 50 kV-4W X-ray tube generator, which employs a Rh anode. The excitation X-ray beam is collimated to a ~1.2 mm spot diameter on the sample surface. The typical energy resolution of the spectrometer is below 135 eV, which is helpful in detecting the asymmetry of the Fe $K\beta$ peak due to the presence of an underlying low-intensity Co $K\alpha$ peak, as is often the case in iron meteoritic samples.

Analysis of the dagger blade was carried out at the Egyptian Museum of Cairo. The XRF head was mounted on a stable tripod equipped with a lateral side arm (60 cm long).

Analysis of meteorites of well-known composition and of certified steel reference materials was carried out in the XGLab laboratory. The XRF head was mounted on a benchtop stand.

For all measurements, the following experimental conditions were used: working distance ~1.4 cm, tube voltage = 50 kV, tube anode current = 80 μA, acquisition time = 120 s.

XRF Data Analysis

The parameters of a model of the shape of the Fe *K*β peak detected by the employed XRF spectrometer were retrieved by using XRF data of a Co-poor steel sample (NIST SRM 1158; Table 1). For this purpose, the Fe *K*β peak was modeled as the sum of a Gaussian and a complementary error function (Jorch and Campbell 1977) (Fig. 3b, red line). In XRF data of samples with detectable Co concentrations, a clear asymmetry of the Fe *K*β peak is visible, induced by the superposition of the close Co *K*α peak. In order to highlight this asymmetry in the XRF dagger spectrum, the right part of the Fe *K*β peak has been fitted with the same model (Fig. 3b, black line).

Estimate of Ni and Co wt% in analysis points of Tutankhamun's dagger has been performed with the following two step-procedure:

1. XRF spectra of all samples were processed to quantify the integrated area (expressed as emission counts per sec) of the detected XRF peaks. We used the PyMca software (Solé et al. 2007), based on a nonlinear least-squares fitting procedure which optimizes zero, gain, noise, and Fano factors for the entire fitting region and for all XRF peaks simultaneously. The background was estimated with the strip background model.
2. Fitted values of the integrated area of Ni (*K*α and *K*β) and Co (*K*α) XRF peaks of reference samples (meteorites of well-known composition and certified steel reference materials) were used for assessing the Ni and Co linearity calibration curves. We employed a robust linear regression model, little affected by outliers, which models the relationship between the wt% composition of the considered element (Ni or Co) and the median value of the related integrated peak area within each sample.

Compositional and class information of a set of 76 iron meteorites with composition similar to Tutankhamun's blade (see Fig. 6) has been provided through access to the Meteorite Information Database (MetBase 7.3; Jörn Koblitz 2015).

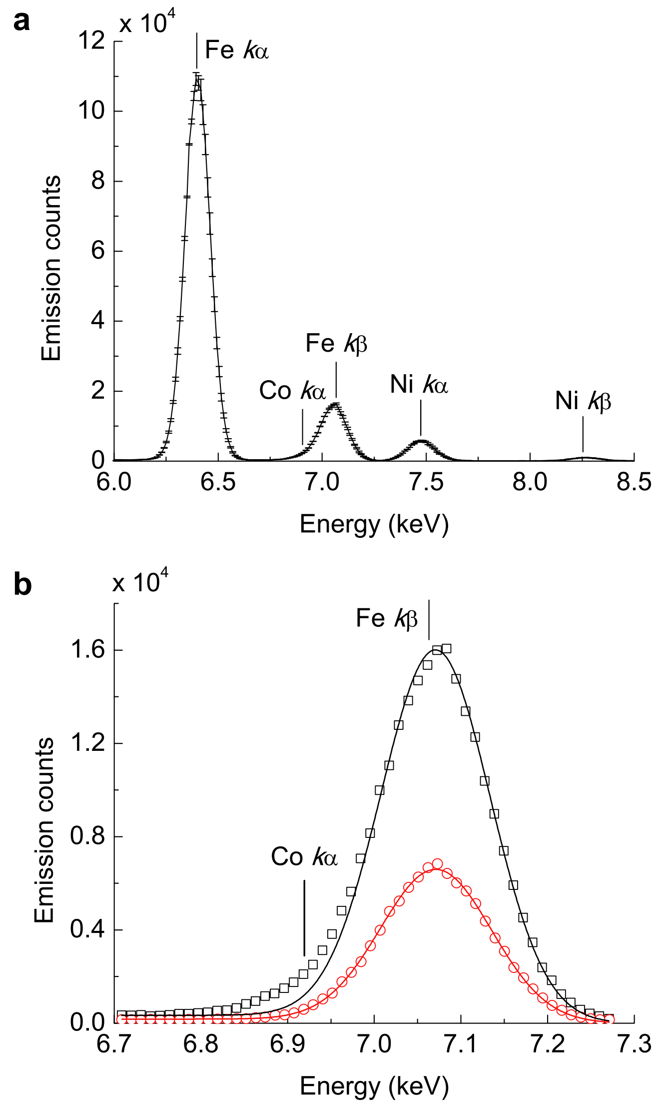

Fig. 3. XRF spectrum of Tutankhamun's dagger blade. a) Median XRF spectrum of Tutankhamun's dagger blade (black line). Vertical error bars depict the interquartile range of the XRF emitted counts. b) Median XRF spectra of the dagger (black squares) and of the Co-poor (0.01 wt%) NIST SRM 1158 steel reference sample (red circles). Each spectrum was fitted with a Gaussian curve peaked at the Fe *K*β line (continuous black and red line), which reveals the asymmetry of the Fe *K*β emission peak in the spectrum of the dagger, namely a shoulder in correspondence of the Co *K*α line.

RESULTS

XRF measurements carried out at the Egyptian Museum of Cairo on two areas of the surface of the dagger blade demonstrate that Fe and Ni are the main bulk constituents (Fig. 3a). The presence of minor concentrations of Co leads to a clear asymmetry in the Fe *K*β emission peak (Fig. 3b).

Quantitative determination of the Ni and Co contents in the dagger was carried out by the external calibration method using XRF data from 11 steel metal standards and 11 iron meteorites of well-known composition (Table 1, Figs. 4 and 5). This allowed the determination of 10.8 ± 0.3 wt% Ni and 0.58 ± 0.04 wt% Co, within a 95% confidence interval (Table 1).

The blade's high Ni content, along with the minor amount of Co and a Ni/Co ratio of ~20, strongly suggests an extraterrestrial origin.

1. The Ni content in the bulk metal of most iron meteorites ranges from 5 wt% to 35 wt%, whereas it never exceeds 4 wt% in historical iron artifacts from terrestrial ores produced before the 19th C (Tylecote 1992).
2. The Ni/Co ratio in the dagger blade is consistent with that of iron meteorites (average Ni/Co = 18 ± 2) (Mittlefehldt et al. 1998), which have preserved the primitive chondritic ratio (~21) (Tagle and Berlin 2008) during planetary differentiation in the early solar system.

Remarkably, a representative set of 76 iron meteorites with a moderately high Ni content (10–12 wt%), i.e., with composition similar to Tutankhamun's blade, have average Co content of 0.57 wt% (±0.08; 1σ) (Fig. 6).

On the sole basis of the Ni and Co contents determined in this work, the meteorite used to fashion the dagger blade cannot be classified into a specific chemical or structural group. Nevertheless, considering the set of 76 iron meteorites mentioned above (Fig. 6), we observe that (1) 25% are ungrouped irons, 22% belong to the IAB complex, 20% to the IID chemical group, 18% to IIIAB, 15% to IIC, IIF, IIIE, IVA; (2) more than 50% have fine (mm scale) or very fine (μm scale) homogeneous structures (e.g., iron meteorites belonging to the ataxite, and fine, finest and plessitic octahedrite structural groups; Fig. 6). Smithing iron meteorites with such homogenous and fine structures are expected to produce a homogeneous structureless iron artifact like the iron blade of Tutankhamun's dagger. Future microstructural analysis of the dagger, if allowed, would provide significant information on the employed manufacturing method.

In order to investigate if known iron meteorites within the ancient Egyptian trade sphere could be linked to the studied blade, we sorted all the known iron meteorites found in the region from the MetBase. Within an area 2000 km in radius arbitrarily centered in

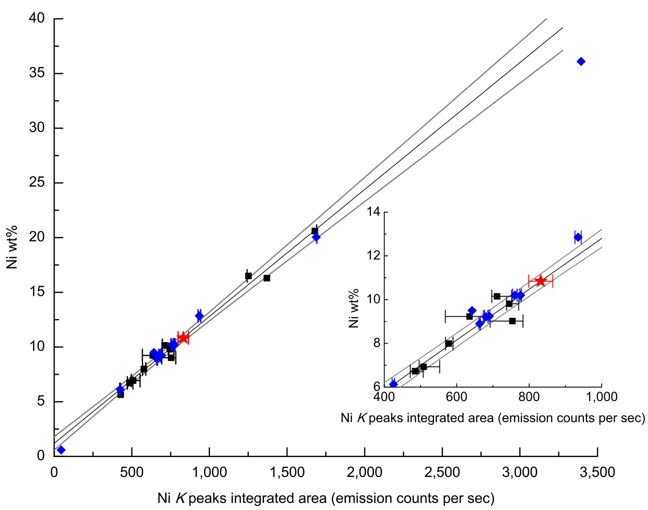

Fig. 4. Ni linearity calibration curve. Calibration curve plot of Ni content (wt%) as a function of the sum of the integrated area of Ni Kα and Kβ peaks (expressed as emission counts per sec). Meteorites and steel reference samples of known composition are shown (black filled squares and blue filled diamonds, respectively). For each sample, the median values of the emission counts are reported, with horizontal error bars depicting the related interquartile range. The retrieved linear regression (R^2 = 0.99) (black continuous line) within a 95% CI (gray continuous lines) is shown. The estimated Ni concentration in Tutankhamun's iron dagger is indicated by the red star. In the inset, a zoomed portion of the graph is shown.

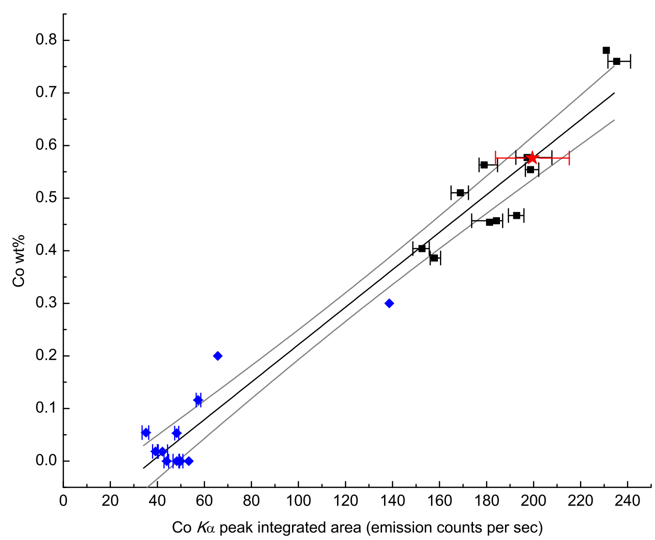

Fig. 5. Co linearity calibration curve. Calibration curve plot of Co content (wt%) as a function of the integrated area of Co Kα peak (expressed as emission counts per sec). Meteorites and steel reference samples of known composition are shown (black filled squares and blue filled diamonds, respectively). For each sample, the median values of the emission counts are reported, with horizontal error bars depicting the related interquartile range. The retrieved linear regression (R^2 = 0.95) (black continuous line) within a 95% CI (gray continuous lines) is shown. The estimated Co concentration in Tutankhamun's iron dagger blade is indicated by the red filled star.

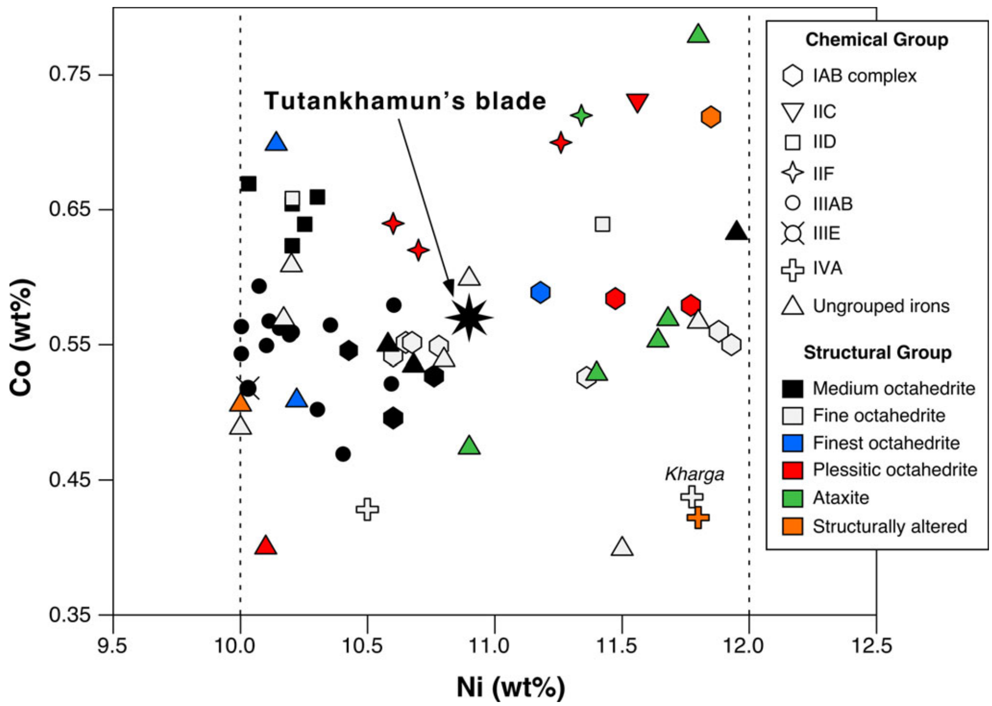

Fig. 6. Co versus Ni diagram for Tutankhamun's iron dagger blade (black star) and for iron meteorites with a moderately high Ni content (10–12 wt%), i.e., with composition similar to the Tutankhamun's blade, sorted by chemical and structural groups.

the Red Sea, Egypt (i.e., extending from central-eastern Sahara to the Arabic Peninsula, Mesopotamia, Iran, and Eastern Mediterranean area), 20 iron meteorite finds are present in the database. Only one, group IVA, the fine octahedrite, named Kharga (Egypt, 31°07′57″N, 25°02′50″E, found 2000, May 8, 1 kg; Grossman and Zipfel 2001), has Ni and Co contents (11.77 wt% and 0.437 wt%, respectively) within 10% of the composition of the studied blade (Fig. 6).

CONCLUSIONS

Recently it has been reported that the most ancient Egyptian iron artifacts, i.e., nine small beads, excavated from a tomb in Gerzeh (Egypt) and dated about 3200 BCE (Stevenson 2009), are made of meteoritic iron, carefully hammered into thin sheets (Johnson et al. 2013; Rehren et al. 2013). Our finding confirms that excavations of important burials, including that of King Tutankhamun, have uncovered pre-Iron Age artifacts of meteoritic origin (Johnson et al. 2013).

As the only two valuable iron artifacts from ancient Egypt so far accurately analyzed are of meteoritic origin, we suggest that ancient Egyptian attributed great value to meteoritic iron for the production of fine ornamental or ceremonial objects up until the 14th C. BCE. Smelting of iron, if any, has likely produced low-quality iron to be forged into precious objects. In this context, the high manufacturing quality of Tutankhamun's dagger blade is evidence of early successful iron smithing in the 14th C. BCE. Indeed, only further in situ, nondestructive compositional analysis of other time-constrained ancient iron artifacts present in world collections, which include the other iron objects discovered in Tutankhamun's tomb, will provide significant insights into the use of meteoritic iron and into the reconstruction of the evolution of the metal working technologies in the Mediterranean.

Finally, our finding provides important insight into the use of the term "iron", quoted in relationship with the sky in Mesopotamian, Hittite, and Egyptian ancient texts (Bjorkman 1973; Waldbaum 1999): beside the hieroglyphic "**bi3**," which already existed before the XIX dynasty with a broad meaning (as "mineral, metal, iron") (Erman and Grapow 1982; Hannig 2003, 2006), a new composite term "**bi3 n pt**," literally translated as "iron of the sky," came into use in the 19th dynasty (13th C. BCE) to describe all types of iron (Bell and Alpher 1969; Erman and Grapow 1982). In the same period, we can note a text at Karnak probably describing a meteorite[3] (Kitchen 1975). The introduction of the new composite term suggests that the ancient Egyptians, in the wake of other ancient

[3]Database Research Unit ISMA-CNR "Egyptian Curses" (PRIN 2009 "The Seven Plagues"): http://webgis.iia.cnr.it/eGIStto/.

people of the Mediterranean area, were aware that these rare chunks of iron fell from the sky already in the 13th C. BCE, anticipating Western culture by more than two millennia.

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SUPPORTING INFORMATION

Additional supporting information may be found in the online version of this article:

Fig. S1. Location of spot analyses on the iron dagger blade: Close-up color image of the iron dagger (Carter no. 256K, JE 61585) with location of the two spot analyses (red circles) ~1.2 mm in diameter performed by XRF spectrometry.