Journal of Archaeological Science: Reports 57 (2024) 104622

Contents lists available at ScienceDirect

Journal of Archaeological Science: Reports

journal homepage: www.elsevier.com/locate/jasrep

Environmental Scanning Electron Microscopy of copper alloy artefacts from unidentified shipwreck sites: Clues to the identity of shipwrecks on Kenn Reef, Coral Sea

Maddy McAllister [a,*], Wendy van Duivenvoorde [b]

[a] James Cook University and Queensland Museum Network, 1 James Cook Drive, Douglas, Queensland 4811, Australia
[b] Flinders University, Sturt Road, Bedford park, South Australia 5042, Australia

A R T I C L E I N F O

Keywords:
Maritime archaeology
Environmental Scanning Electron Microscope
Copper alloys
Archaeometry
Shipwrecks

A B S T R A C T

This study investigates the use of Environmental Scanning Electron Microscopes (ESEM) as a tool for identification of historic shipwrecks. Copper alloy fasteners from unidentified shipwreck on Kenn Reef in the Coral Sea, were sampled and analysed to determine the chemical composition of each artefact. When combining this data with known evolution of copper alloying for wooden ship construction, historical and archaeological data, the results provided valuable insight into the possible date range for these wrecks. Specifically, comparison to known wrecks in the vicinity of Kenn Reef could be linked to these previously unidentified sites. Results are promising and ESEM analyses proved to provide valuable, reliable data, yet also highlighted limitations and areas for further investigation and research.

1. Introduction

Over 1,400 ship and aircraft wrecks lie scattered across the Queensland coastline, the majority wrecked along the Great Barrier Reef (GBR) ([Queensland] Department of Environment and Science 2023). While some are infamous, the less significant sites are listed as unidentified, known only by association to the reefs they are located on. Over 8,000 objects from approximately 28 shipwreck sites located in Queensland and the Coral Sea are housed in the Queensland State Maritime Archaeology Collection, at the Queensland Museum, Tropics (QMT). Of these, at least twelve are mystery shipwrecks—their names and dates of sinking remain unknown.

The dynamic and turbulent environments of the Great Barrier Reef and Coral Sea create unstable conservation of sites and often the only material left of historic shipwrecks are metal components, fragmented glass and ceramics. A combination of archaeological investigations by the Queensland Museum and material donations resulted in a large collection of shipwreck material from this reef. While the archaeological material is of known provenance within the reef itself, the donated material is less certain. In addition, the identity of the shipwreck sites on Kenn Reef are largely unknown. This paper reports the results of Environmental Scanning Electron Microscope (ESEM) composition analyses of copper alloy fastener and sheathing artefacts from unidentified shipwreck sites on Kenn Reef (van Duivenvoorde and McAllister, 2021) and explores ESEM as a valid tool for shipwreck identification. Results of these analyses will allow a refined understanding of when these vessels were built and contribute to narrowing down the identity of the wrecks. The method described in this paper requires minimal destructive sampling and results give a strong indication of data that greatly improves knowledge of these shipwreck sites.

1.1. Copper alloys and shipbuilding over time

Copper sheathing originated as a superior sheathing to lead, wood or leather, yet consequently reacted badly with iron fastenings—galvanic action (Bingeman et al., 2000:222; Harris, 1996:553; McAllister, 2012:40; McCarthy, 2005:103; Van Duivenvoorde, 2012,2015). As shipbuilders began experimenting with copper fastenings, pure copper (99 % and more) was suitable, yet expensive and overly soft before hammered and annealed (McCarthy, 2005:102; Van Duivenvoorde, 2014). Consequently, combinations of copper and zinc alloys, called ‘mixed metal’ or commonly known as brass, combined the malleability of copper with the reduction of corrosion provided by zinc (McAllister, 2012:40, McCarthy, 2005:103). For example, the patenting of a copper alloy renowned as ‘Muntz metal’ in 1832 serves as an approximate date marker for the implementation of fasteners comprising of 60 % Copper

* Corresponding author.
E-mail address: madeline.mcallister@jcu.edu.au (M. McAllister).

https://doi.org/10.1016/j.jasrep.2024.104622
Received 24 September 2023; Received in revised form 14 April 2024; Accepted 27 May 2024
Available online 10 June 2024
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and 40 % zinc (Bingeman et al., 2000:224; Flick, 1975:74; McCarthy, 2005:115). In fact, under George Muntz's patent no. 6325, the proportions could be anywhere between 50–63 % copper and 37–50 % zinc, yet the 60:40 ratio was preferred (Patent No. 6325, 1832). Significantly, historical evidence notes that Muntz struggled to break into the sheathing market for some time after his patent was approved (Flick, 1975:86). In fact, in 1837 only 50 ships were sheathed with Muntz metal in the United Kingdom, increasing to 400 by 1840 (Flick, 1975:77, Staniforth, 1985:27). In addition, precise formulation of copper alloys was always difficult to achieve sure to the much more volatile zinc (McAllister, 2012:410; Van Duivenvoorde et al., 2023), hence, variations have been given at 60–62 % copper and 30–38 % zinc (Vickers, 1923:425) and modern metallurgists assign 59–63 % copper, 36.63–40.63 % zinc, 0.30 % lead and 0.07 % iron (Kundig and Weed, 2015:128). Other historical patents exist, such as William Foster and Co's that is similar, yet with different compositions and included within this study to provide additional comparative discussion of the results (Flick, 1975; Van Duivenvoorde, 2019b, 2020).

Previous research indicated some limitations in using the chemical composition of copper alloys from shipwreck sites to determine age of a vessel. For example, McCarthy (2005:136) highlighted previous studies where copper fastenings recovered from shipwrecks without context (i.e. not found within their parent timber) had to be carefully scrutinised before inclusion into analysis (Larn et al., 1974; Philpin, 2024:24; Stanbury, 1994: 103). Stanbury (1994:14) went further to indicate that given the rapid technological advances in copper bolt manufacture archaeological dating of a vessel based on compositional data from copper alloy bolts is not a reliable indicator. In addition, the potential impact of recycled ship's fastenings being used in construction and/or repairs of vessels adds another layer of possible error. Recycling and reuse of metals from wooden ships is a well-known and recognised standard within global shipbuilding (Delgado, 2009; McCarthy, 2005; Staniforth and Richards, 2015). Vessels at the end of their use/lifetime were often discarded in either shallow water or burned to facilitate recovery of copper fastenings with ease (McCarthy, 2005). Consequently, we should expect to have some level of copper alloy composition variation even within a site that has reliable context, and this factor is acknowledged within the results of this study. Despite these limitations in context and reuse of copper fastenings, this study aims to reintroduce chemical composition analyses of copper alloys as a potential tool that may indicate age of a shipwreck, particularly for sites that have little else remaining and from which this valuable data may lead to possible identification of a wreck.

Initial research into chemical composition of copper alloys by Ian Macleod (1987) serves as a baseline indicator for the percentage of copper alloy fasteners used in known wooden shipwrecks across

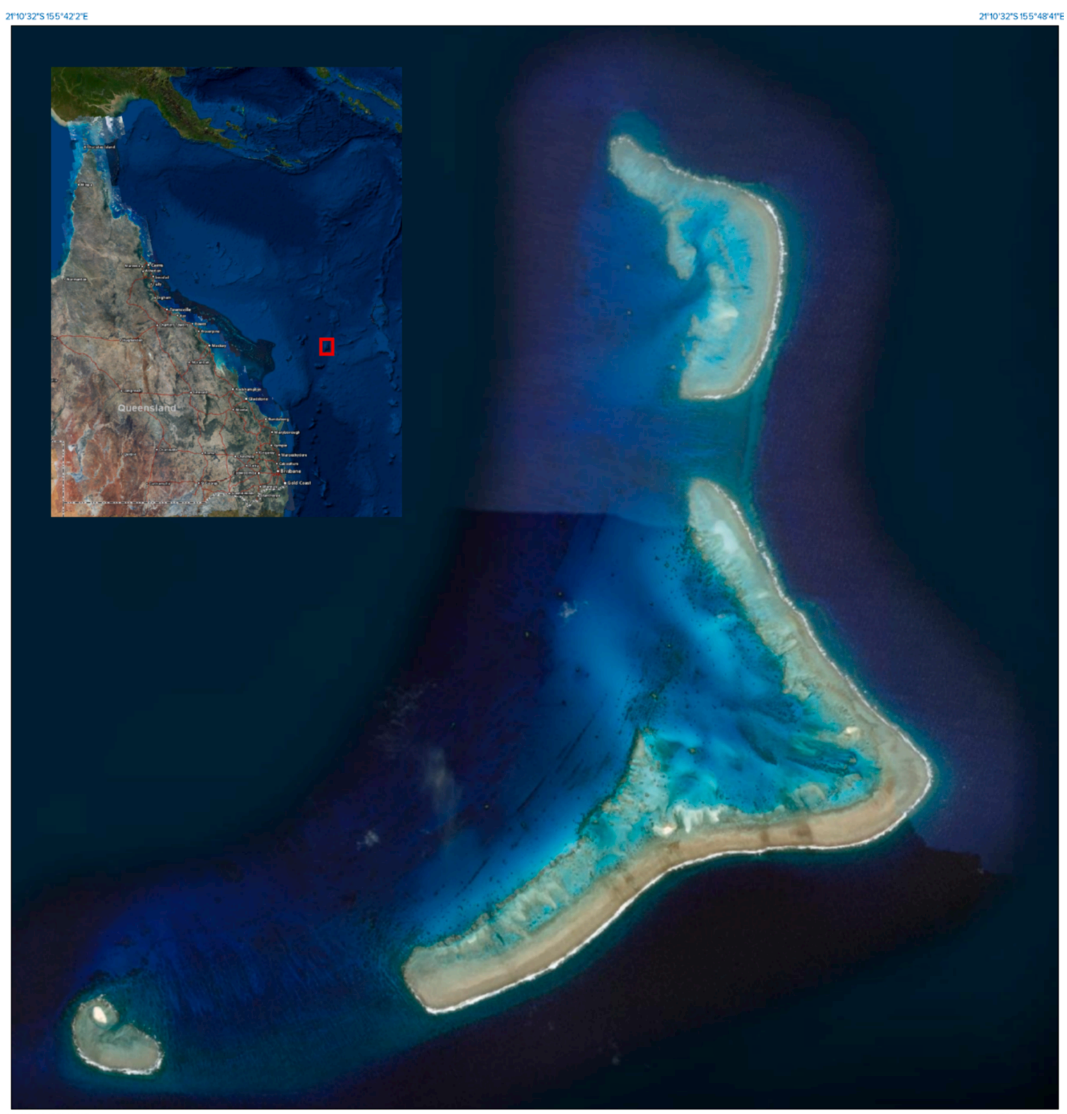

Fig. 1. Satellite image of Kenn Reef and insert of the Queensland coast (State of Queensland 2021, adapted by M. McAllister).

Australia. Higher levels of copper generally indicated ships built pre-1830 s. Additional analyses provide further comparative ships and dates they were built (Bennett, 2021,2023; Bingeman, 2018; Chandrakasekaran, 2019; Hunter et al., 2023; McAllister, 2012; McCarthy, 2005; O'Guiness et al., 2010; Philpin, 2024; Philpin et al., 2021; Stanbury et al., 2015; Stanbury, 1994; Van Duivenvoorde 2019a, 2019b, 2020; Van Duivenvoorde et al., 2023; Villalobos, 2020; Zapor, 2020).

1.2. Kenn Reef shipwrecks

One area, Kenn Reef in the Coral Sea, proved to be a magnet for shipwrecks in the nineteenth century. Kenn Reef is a coral seamount system located at the outer edge of the Great Barrier Reef in the Coral Sea. This reef sits approximately 520 km northeast of Bundaberg, Queensland and the nearest neighbouring geographical feature, Wreck Reef, is located 100 km to the southwest. Kenn Reef is in the shape of a backwards 'L' or boot. It stretches about 15 km in length and 8 km in width (Fig. 1). In total, at least eight vessels are suspected of wrecking on this reef system (Table 1).

Due to the remote location of Kenn Reef, archaeological investigation of the located shipwreck sites is limited. In 1987, the Maritime Archaeology Department of the Queensland Museum investigated Kenn Reef to locate and assess the archaeological potential of the shipwrecks. The Museum recorded most of the sites located at Kenn Reef and recovered various artefact material. In particular, Kenn Reef 4 was identified as the remains of *Jenny Lind* (1850) and Kenn Reef 6, on the most north-eastern corner of the reef as *Bona Vista* (1828) (Queensland Museum file/Kenn Reef 1/2, 1987).

In 2017, a joint expedition by the Australian National Maritime Museum (ANMM) and Silent World Foundation aimed to survey known shipwreck locations at Kenn Reef and undertake magnetometer surveys to find new sites (Hosty et al., 2017:20). The 2017 surveys confirmed Delaney and Batley's identification of *Jenny Lind*. Of relevance to this research, the 2017 team relocated Kenn Reef 1 (KR1), Kenn Reef 2 (KR2), *Jenny Lind* (KR4) and Kenn Reef 7 (KR7) yet they were unable to relocate Kenn Reef 3 (KR3), Kenn Reef 5 (KR5) and *Bona Vista* (KR6).

Table 1
List of vessels wrecked or possibly wrecked on Kenn Reef.

Vessel name	Type	Tonnage	Built: Year (Country)	Wrecked: Year	Reference
Bona Vista	Brig	237	1825 (UK)	1828	The Tasmanian, 1828:2, Stone, 2006
Jenny Lind	Barque	484	1847 (Canada)	1850	The Maitland Mercury and Hunter River General Advertiser, 1850:4, Stone, 2006
Delta	Frigate	930	1839 (Netherlands)	1854	Lloyd's, 1853 Hosty et al., 2017
Hester	Ship	840	1833 (Netherlands)	1854	Stone, 2006, Sydney Morning Herald, 21 June 1854: 4
Doelwijk	Ship	740	1850 (Netherlands)	1854	Stone, 2006, Sydney Morning Herald, 21 June 1854: 4
Lion	Ship	298	1823 (America)	1856	Account book of Lion (ship) 1846.
Rodney	Barque	877	1850 (UK)	1858	The Age, 12 October 1858: 4, Stone, 2006
Oliver van Noort	Ship	807	1851 (Netherlands)	1858	Loney, 1980: 93

1.3. The 1993 amnesty

Australia's underwater cultural heritage is managed and protected under the Commonwealth *Australian Underwater Cultural Heritage* Act (2018). Preceding this legislation, shipwrecks were protected under the *Historic Shipwrecks* Act (1976). To acknowledge the long-held interest of recreational divers and shipwrecks, an amnesty occurred in 1993 through which people could come forward with shipwreck material in their possession and hand it over to the relevant authority without reprimand (Rodrigues, 2009). During this amnesty, renowned underwater photographers and ocean activists, Ron and Valerie Taylor, donated a collection of copper alloy shipwreck material to the Queensland Museum. The collection comprised a large amount of copper alloy fasteners and fittings as well as a large pintle and a spectacle plate (Queensland Museum File MA 15/8). Correspondence between the Museum and Ron Taylor indicated that the material was collected in the early 1960 s in approximately two metres of water and approximately two kilometres from Observatory Cay (Queensland Museum File MA15/8). Furthermore, correspondence confirmed this material most likely originated from KR3, although Taylor was unable to confirm it. Given the significant collection of material, particularly the pintle and spectacle plate, incorporating the material into this research is essential as it could reveal new information about KR3 and assist with further identification.

While this paper focuses on the 1987 material collected by the Museum and comparisons with the donated Taylor collection, some relevant findings of the 2017 survey are added for additional confirmation and comparison. The related sites, KR 1, KR 2, KR 3, and KR 7 (Fig. 2) are outlined below to provide background for the ESEM results. Remaining sites are excluded from this study as they are either identified or lack any copper alloy materials recovered from them for analysis. Although no copper alloy material was recovered from KR2, an outline is included based on the possible relation to KR7.

1.4. Kenn Reef 1

KR1 is situated 1200 m southwest of Observatory Cay at the reef's edge. The site is spread over 50 m long in an east to west oriented fan-like scatter of structural material (including anchors, mast components, chains, hawsepipes, wood and copper alloys etc.). At low tide, most of the features sit above water. Three admiralty pattern anchors were recorded at the site in 1987, the smallest closest to the reef edge and the other two lie close together at the farthest inshore edge of the artefact scatter. Notably, there are two iron masts located within KR1, measuring seven and ten metres in length. Both masts have the support cheeks, and one has an intact mast cap, although Delaney and Bately fail to clarify if it is the longer or shorter one (1987:9). In addition, there is a length of stud link chain with hawsepipes running from the inshore anchors to the reef's edge. Some other concreted, yet unidentified material is also visible on the site. The 1987 team recovered four objects: a copper alloy sheathing fragment, a clenched copper alloy bolt with washer and wood and two fragments of wood with bolt holes. As only two vessels wrecked on Kenn Reef are known to have iron masts, it is likely that KR 1 is either *Doelwijk* or *Olivier van Noord.* In 2017, ANMM hypothesised that KR1 was *Doelwijk* as it matched historical accounts of the wrecking event, along with features found on the site (Hosty et al., 2017).

1.5. Kenn Reef 2

This site is located approximately 500 m south of Observation Cay on the reef edge. KR2 is a relatively large site, with material stretching

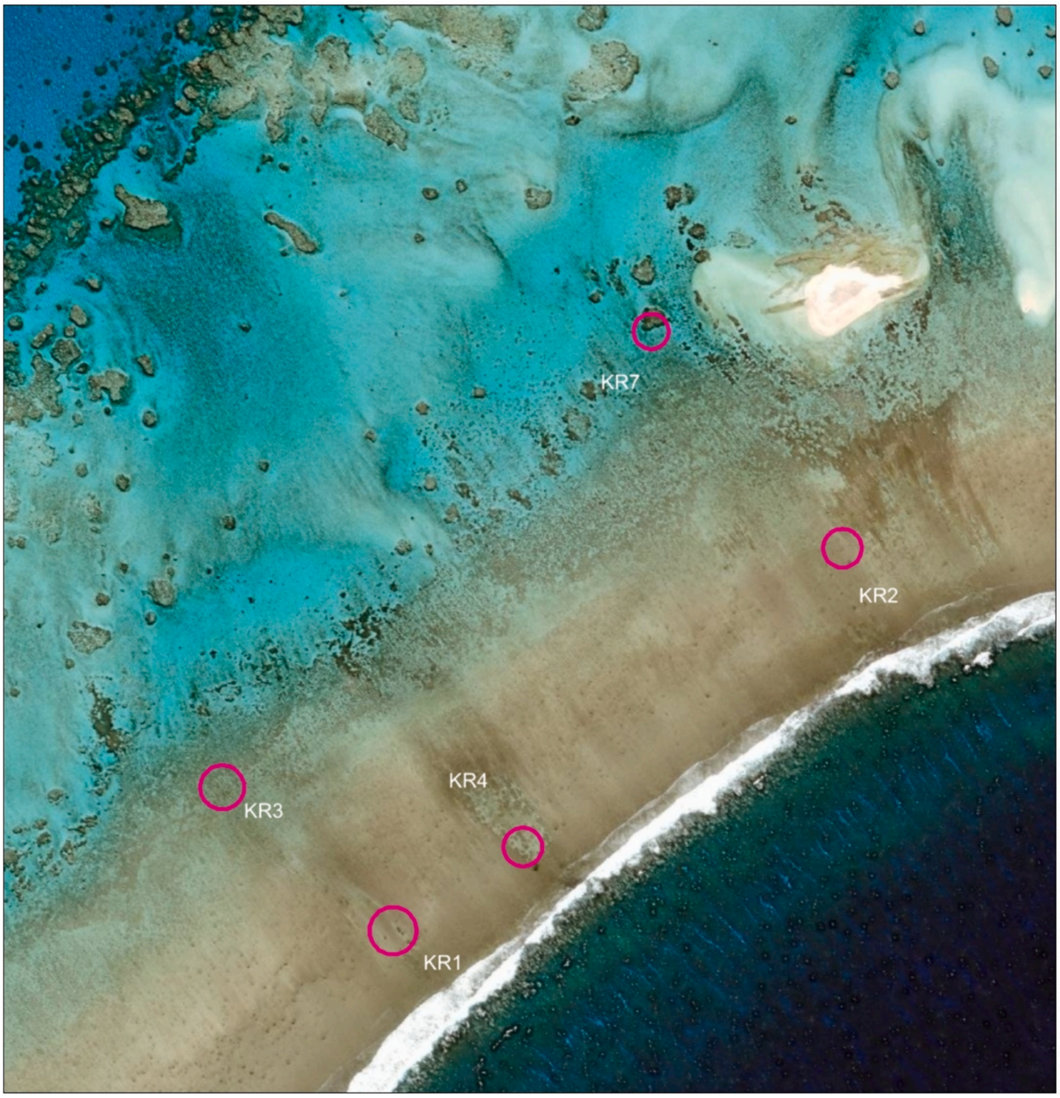

Fig. 2. Close-up of the Kenn Reef sites relevant to this study (KR1, KR2, KR3, KR4 and KR7 (State of Queensland 2021, adapted by McAllister 2021).

across a 100 m long swathe running north westerly from the edge. Prominent features of the site include a winch, a 3.2 m long iron shaft, a gear wheel and three round-armed, admiralty pattern anchors dominate the site. No copper material was recovered from this site. Although Delaney and Bately could not conclusively identify this site in 1987, Hosty et al., (2017:80) theorised it could be *Hester* based on the site's location in relation to *Doelwijk* in historical accounts of the wrecking event.

1.6. Kenn Reef 3

Unlike sites KR1 and KR2, KR3 is located inside the lagoon on the eastern side of the reef. It lies 800 m southwest of Observatory Cay on the inner edge of the reef flat in approximately four metres of water. A mix of material was found to stretch across a nine-metre-long concentrated line running at a north easterly direction. Artefacts identified include iron structural remains (iron knees measuring 1200 mm x 1200 mm), copper alloy bolts, fragments of sheathing and bricks. Delaney and Batley (1987:7) hypothesised that KR3 could comprise of material from KR1 and KR4 as the site sits in line with these sites and prevailing winds and currents could have pushed material across the reef surface. The site could not be relocated by Delaney in 2003, nor in 2017 (Hosty et al., 2017:18). In 1987 two copper alloy sheathing fragments, two copper alloy nails (one with wood attached), two brick fragments, a square black bottle base and a fragment of lead piping were collected. In addition, Delaney later hypothesised that this could be the site of the American whaler *Lion,* generally thought to have wrecked on Wreck Reef—farther south. Delaney suggested *Lion* was possibly at KR3 instead as it was coming from the North and heading southeast—unlike the other known wrecks on the reef (Queensland Museum file/Kenn Reef 2/2, 1987).

1.7. Kenn Reef 7

Like KR 3, this site sits within the lagoon. Kenn Reef 7 (KR7) is situated approximately 200 m west of Observatory Cay. Delaney and Bately (1987:12) hypothesised that the wreckage was likely to be associated with KR 2 as it could easily have washed inshore from the reef edge. Another reason for this suggestion is the lack of larger features at KR 7 (for example no anchors, masts or ballast). Material identified at KR7 included: fragments of sheathing, broken earthenware storage jars, broken glass bottles, a copper alloy door lock, a pulley sheave and a copper alloy bolt. Objects recovered by the 1987 team include: an earthenware fragments (neck, base and wall), three fragments of copper alloy sheathing, the door lock, the wooden pulley sheave, and a copper alloy bolt with wood attached. The 2017 expedition's discovery of material at KR7 is significant for this research. The addition of iron frames, anchor chain, winch/capstan components and two hawsepipes suggest that it could be a separate site to KR2. Primarily based on the presence of additional hawsepipes. Despite tentatively assigning a tonnage (150 tons) from the dimensions of this material, no further identification could be made from the site or artefacts remaining there (Hosty et al., 2017:85).

2. Materials and methods

2.1. Sampling

A selection of copper alloy artefacts was identified as potentially providing us with compositional information. Chosen artefacts met the following criteria: good condition allowing for a reliable sample; known context of where they came from within Kenn Reef and an identifiable artefact (for example sheathing or a large bolt). In addition, several artefacts from the Taylor donation were selected as comparable samples to provide more information and potentially determine provenance and matching to known sites. Fourteen artefacts were sampled in total ranging from spikes, bolts, and sheathing to parts of the rudder assemblage (fittings) (Table 2).

The process of preparing the sheathing samples (MA2200, MA2204, and MA2213) involved embedding a small portion of each sample in phenolic mounting resin (Struers MultiFast) for general use. This was done by adding the resin to a Struers CitoPress-10 hot mounting machine and allowing it to set. The specimens underwent resin mounting using the "cold-mounting" method. Subsequently, they were ground and polished using different polishing slurries on a polishing disk in a 'Struers automatic metall3. ographic polisher'. The final polishing step utilized colloidal silica, resulting in mirror-like surfaces free of scratches (Kurdi et al., 2024:4). (Fig. 3). To ensure minimal contamination, a few milligrams of material were extracted from each sample by drilling, using a brand-new titanium drill bit (2 mm) to obtain uncorroded metal samples. A new, unused drill bit was used for each sample and titanium was selected as the drill material as it is foreign to historic alloys, much harder and did not 'add' to the sample. These drilled samples were then placed on a 12 mm aluminium stub using carbon tape tabs as part of the sample preparation process (Fig. 4).

2.2. Environmental Scanning Electron Microscope analysis

Samples from the Kenn Reef collection underwent analysis at Adelaide Microscopy in South Australia using an FEI Quanta 450 FEG Environmental Scanning Electron Microscope (ESEM). The FEI Quanta 450, which is a High-Resolution Field Emission Scanning Electron Microscope, was utilised to image and examine surface topography, capture backscattered electron images, and determine the elemental composition of the samples through x-ray detection using a Silicon Drift Detector Energy Dispersive Spectroscopy (SDD EDS) detector.

The FEI Quanta 450 with SDD EDS detector enables a semi-quantitative analytical approach for determining the elemental composition in specific areas or spots. Since this analysis method focuses on localised testing, it may not represent the composition of the entire sample. To ensure representative results, three characteristic areas per sample were tested whenever possible. These areas were chosen based on their display of solid metal and absence of noticeable surface corrosion. During data acquisition, the following ESEM settings were applied: High-Vacuum, Kilovoltage: kV 20, Element Normalized, SEC table: default, standardless. The time per sample analysis was automated.

For all samples mounted on aluminium stubs, aluminium (Al) was intentionally excluded from the analysis to avoid interference with the EDS detector probe, as it could yield a false positive reading for aluminium. The same applies to carbon (C) as these samples were taped to carbon tape. Carbon inclusions were however visibly present on the polished surfaces of the sheathing samples (refer to Table 4 and Fig. 5). Carbon is a known corrosion product that can appear as isolated inclusions a few millimetres below the surface (pers. comm. Animesh Bashak, Adelaide Microscopy). It must be noted that historical records from the nineteenth century indicate that charcoal was occasionally deliberately added to copper as part of the smelting process (Bennett 2021:293–294, Marr, 2006). For the purpose of this study, carbon was excluded to primarily focus on the composition and metal ratios in alloys.

3. Results & discussion

3.1. Kenn Reef 1

Only two copper alloy artefacts were recovered from KR1, a fragment of copper alloy sheathing (MA2200) and a large bolt with washer (MA2201). The sheathing (MA2200) tested positive for the following elements: Cu (copper) and Zn (zinc). To assess the composition of every sheathing sample, trace elements such as arsenic (As), silver (Au), bismuth (Bi), iron (Fe), lead (Pb), nickel (Ni), antimony (Sb), and tin (Sn) were manually added to the spectra and tested, as they are known to be present in small quantities in copper alloys (McLeod 1987). This inclusion of trace elements also helps determine if iron, lead, and tin are present in concentrations higher than what would be expected for trace elements, as they were often intentionally added to copper alloys used for ship's hull sheathing. Specifically, the sheathing samples contain some lead, which can be clearly observed in the micrographs as small white specks dispersed throughout the samples (Figs. 5, 8-9). Spot analysis confirms the presence of lead in the copper-zinc alloy, although the weight percentages are low and range between 0.15 % and 0.41 %. These low weight percentages indicate that the lead is present as a trace element.

Results indicate that the sheathing (MA2200) is made of a yellow metal like Muntz patents with an average copper-zinc ratio of 63:37 (Table 3) (Chandrasekaran, 2019; Flick, 1975; McAllister, 2012; Van Duivenvoorde 2019b, 2020; Van Duivenvoorde et al., 2023). The bolt from KR1 (MA2201) showed slightly higher copper ratios, it has an average weighted percentage of 66.25 % copper and 31.90 % zinc, with small amount of lead, iron and tin that are low enough to be considered trace elements. This bolt is also similar in composition to yellow metal like Muntz patents.

3.2. Kenn Reef 3

A copper alloy spike (MA2205) and a fragment of copper alloy sheathing (MA2204) from KR3 were analysed. The sheathing (MA2204) tested positive for the following elements: Cu (copper) and Zn (zinc). MA2204 was made of a yellow metal similar to Muntz and Williams Foster & Co patents with an average weight percentage of 64.31 % copper and 35.04 % zinc (Table 4) (Chandrasekaran, 2019; McAllister, 2012; Van Duivenvoorde 2019b, 2020). The spike (MA2205) tested positive for the following elements: Cu (copper) and Zn (zinc). MA2205 has an average weight percentage of 64.98 % copper and 31.33 % zinc. Both artefacts have similar copper to zinc ratios and with trace elements of lead, iron and tin. Consequently, we estimate that the fasteners were made of a yellow metal like Muntz and Williams Foster & Co patents with a copper zinc ratio of about 66–70 % copper and 30–34 % zinc.

When compared to other known shipwrecks, these results are similar to German-built *Gemma* (built in 1868) (MacLeod, 1987:283) and British-built *Amazon* (built in 1855). Results place the construction of this vessel to possibly the mid-nineteenth century. Here it is significant to return to the potential for recycled and reused copper fasteners (see 1.1) (McCarthy, 2005). The authors endeavoured to sample first large bolts that would likely be related to internal structures such as keel bolts, and therefore less likely to have been removed and repaired over time. However, we acknowledge that this does not rule out use of recycled fasteners in initial construction of a vessel and all possible identifications

Table 2
Samples taken and relevant information for copper alloy analyses.

Site	Registration No.	Artefact type	Dimensions (mm) & Weight (kg)	Image
KR1	MA2200	Sheathing	L. 140 mm W. 60 mm Th. 1 mm 0.04 kg	
KR1	MA2201	Bolt with washer, broken	L. 345 mm Diam. 25.5 mm Wash. Diam. 41 mm 1.14 kg	
KR3	MA2204	Sheathing, folded/crumpled	L. 170 mm W. 50 mm Th. 5 mm 0.08 kg	
KR3	MA2205	Spike	L. 182 mm W. 150 mm Th. 100 mm Head Diam. 22 mm 0.18 kg	
KR7	MA2213	Sheathing	L. 60 mm W. 20 mm Th. 3 mm 0.02 kg	
KR7	MA2217	Bolt with wood attached, whole	L. 325 mm Diam. 17.5 mm 0.64 kg	
Unknown: R&V Taylor donation	MA2220	Pintle	L. 1040 mm W. 380 mm H. 440 mm 80–100 kg	
Unknown: R&V Taylor donation	MA2221	Spectacle plate	L. 640 mm W. 480 mm H. 140 mm 60–80 kg	
Unknown: R&V Taylor donation	MA2222	Possible rudder pivot	L. 335 mm Th. 120 mm W. 90 mm 23.44 kg	

(*continued on next page*)

Table 2 (*continued*)

Site	Registration No.	Artefact type	Dimensions (mm) & Weight (kg)	Image
Unknown: R&V Taylor donation	MA2225	Strap – with decoration	L. 480 mm W. 46 mm Th. 5 mm 0.98 kg	
Unknown: R&V Taylor donation	MA2234	Bolt – broken	L. 360 mm Diam. 34 mm 2.94 kg	
Unknown: R&V Taylor donation	MA2248	Bolt – complete both ends hammered	L. 460 mm Head Diam. 36 mm Diam. 28 mm 2.3 kg	
Unknown: R&V Taylor donation	MA2252	Long bolt/keel. Complete: Hammered end and pointed end.	L. 970 mm Head Diam. 35 mm Diam. 26 mm 3.78 kg	
Unknown: R&V Taylor donation	MA2260	Complete through-bolt with clinch	L. 535 mm Head Diam. 28 mm Washer Diam. 45 mm Diam. 22 mm 2.48 kg	

Fig. 3. Kenn Reef ships' hull sheathing samples embedded in black-coloured resin mount after polishing. Photograph by W. van Duivenvoorde.

from this study have the stipulation that further research is needed to confirm these results.

Additionally, results are similar enough to KR1 that we can confirm the two sites are likely one larger wreck site. In addition, Delaney and Batley's, 1987 (QM file Kenn Reef 1/2) field notes indicate that ballast bricks continue in a scattered manner from KR1 to KR3. Iron frames at

Fig. 4. Kenn Reef drill shavings mounted on aluminium stubs with carbon tape. Photograph by W. van Duivenvoorde.

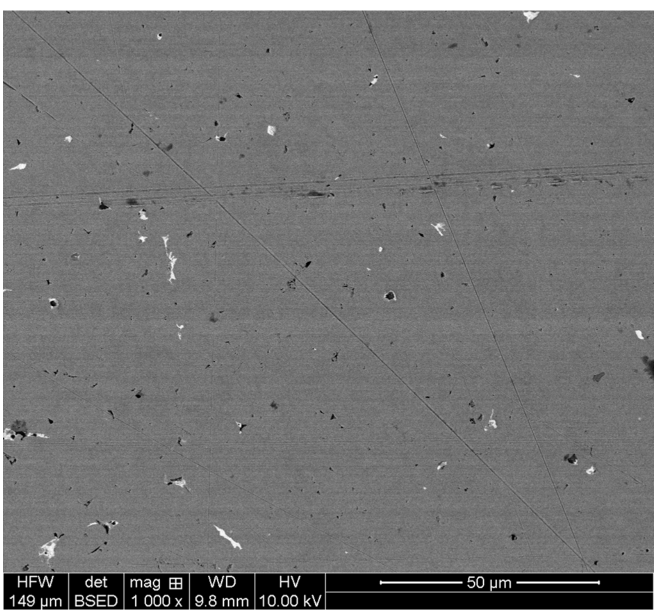

Fig. 5. Testing area on sheathing fragment, MA2200, showing copper alloy surface with small inclusions of lead (white in colour) and carbon (black in colour) Image: A. Basak, W. van Duivenvoorde.

Table 3
ESEM results of KR1 samples.

Artefact	Cu Wt % average	Zn Wt % average	Pb Wt % average	Cu:Zn average ratio
MA2200 Sheathing	62.52	35.19	NA (trace)	63:37
MA2201 Bolt	66.25	31.90	1.03	67:33

Table 4
ESEM results of KR3 samples.

Artefacts	Cu Wt % average	Zn Wt % average	Cu:Zn ratio average
MA 2204 sheathing	64.31	35.04	65:35
MA 2205 spike	64.98	31.33	68:32

KR3 measured 1200 mm x 1200 mm, as did iron frames at KR1 (QM file Kenn Reef 1/2) and the lack of mechanical features such as a windlass, on KR3 also indicate that it may not be a complete site (QM file Ken reef 2/2). Furthermore, the geographical location of KR3 – directly behind KR1 – on the lee side of the reef and in a likely pattern of dispersion further strengthens the argument that KR3 is the farthest extremity of KR1, or *Doelwijk*.

3.3. *Kenn Reef 7*

Sheathing recovered from KR7 (MA2213), tested positive for copper and zinc. The sheathing (MA2213) sample does contain some lead as can be seen clearly in the micrographs as little white specks spread throughout the samples (similar to MA2200 (Figs. 5-6)). The presence of lead in the copper-zinc alloy is confirmed via spot analysis on the white specks, but their weight percentages are very low and vary between 0.15 and 0.41 %. Such low weight percentages indicate that the lead is only present as a trace element. Results indicate that the sheathing was made of a yellow metal similar to Muntz and Williams Foster & Co patents with a copper zinc ratio of about 64–65 % copper and 35–36 % zinc (Table 5) (Chandrasekaran, 2019; Van Duivenvoorde 2019b, 2020, 2023).

In comparison, the bolt (MA2217) is made with pure copper with an average weighted percentage of 99.20 %. The copper itself is quite pure, not alloyed with zinc and contains little or no lead and no iron. This bolt likely came from a vessel built prior to 1832. It closely matches similar copper alloy bolts from other late eighteenth- and early nineteenth-century shipwrecks such as *South Australian* (built in 1819, wrecked in 1837 in South Australia), copper sheathing from the Dutch East India Company (VOC) shipwreck *Zeewijk* (built in 1725, wrecked in Western Australia in 1727), the French built *Lively* (built 1765, wrecked in 1806/

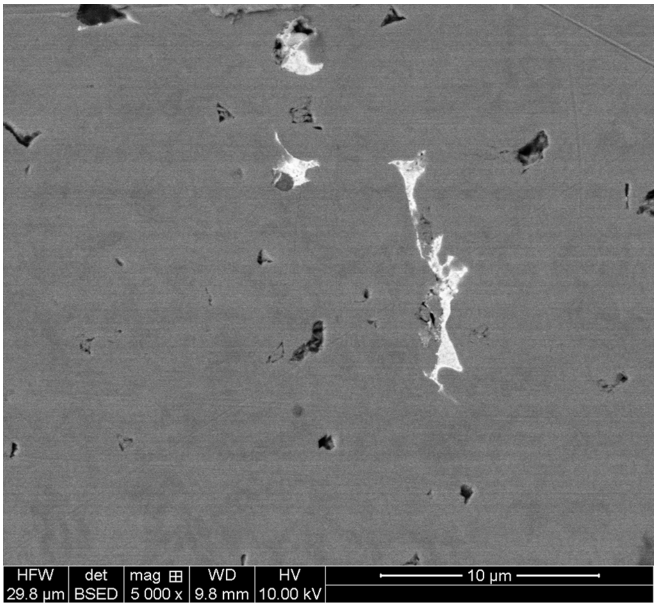

Fig. 6. Micrograph of sheathing sample showing lead inclusions as analysed, MA2200. Image: A. Basak, W. van Duivenvoorde.

7), and American built *Rapid* (built 1809, wrecked in Western Australia 1811) (Hunter et al., 2023; MacLeod, 1987:282; Van Duivenvoorde 2019a; Zapor, 2020). These results suggest that KR7 was a vessel built in the early nineteenth century yet wrecked in the mid-nineteenth century.

In 1987, Delaney and Batley (1987:11) theorised that KR7 was wash over from KR2. Unfortunately, no copper alloy artefacts were retrieved from KR2 to compare with KR7. However, substantial research completed by Hosty et al., (2017:85) on new structural material located on KR7, suggest that KR7 is indeed an individual site. Hosty et al. (2017) compare dimensions of hawsepipes, anchor chains, iron frames they determined KR7 is a separate vessel of approximately 150 tons. The presence of iron riders at KR7 adds additional interesting conjecture as iron was included in the lower vessel construction as riders were developed to "…support hold beams and to give additional strength to the lower hull." (Stammers, 2001:119). Although iron knees, stanchions, breasthooks and crutches were commonplace in newly built British ships by 1810 (Steel, 1823), they were increasingly employed for the construction of larger ships throughout the following decades. This was partly due to the decrease in raw resources for shipbuilding in Britain and the increased strength and compactness provided by iron (Stammers, 2001:115). In comparison, American shipbuilders had no resourcing issues for timber and the inclusion of iron frames in American

Table 5
ESEM results of KR7 samples.

Artefacts	Cu Wt % average	Zn Wt % average	Pb Wt % average	Cu:Zn average ratio
MA2213 Sheathing	64.06	35.14	NA (trace)	64:36
MA2217 Bolt	99.20	NA (trace)	NA (trace)	NA

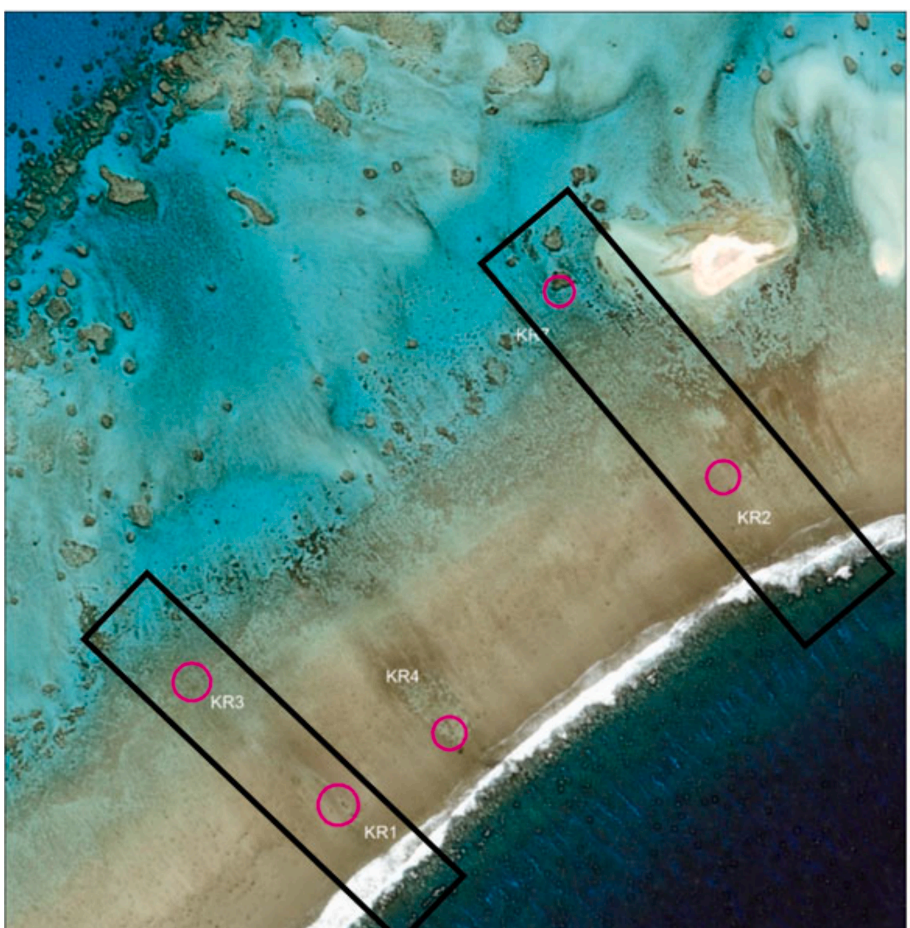

Fig. 7. Satellite imagery of the wrecksite with theoretical dispersion from primary wrecksite to other areas (satellite image © State of Queensland 2021, adapted by McAllister 2022).

shipbuilding came at a much later date (Crothers, 1997; Stammers, 2001:116).

In summary, the results of copper alloy fastener and fittings analysis correspond with previous archaeological work. KR7 matches known vessels with pure copper fasteners built between 1765 to the 1830 s (MacLeod, 1987, Van Duivenvoorde 2019b, 2020a). However, the inclusion of iron riders located on KR1 most likely suggests a vessel constructed from 1810 onwards, if it originates from Europe (Seppings 1814; Stammers, 2001:115, Steel, 1822). The results could imply that it is still *Hester* (built in 1833, wrecked 1854), or possibly *Lion* (built in 1823, wrecked 1856). There is still the possibility at parts of *Hester* washing over onto KR7 given the geological formation of Kenn Reef and impacts of currents and swell from the southern side (Fig. 9).

3.4. The Taylor material

Analysis of the material donated by the Taylors resulted in two distinct groups, the bolt fasteners and the rudder assembly components. Four bolts were sampled from this collection – MA2248, MA2234, MA2252 and MA2260. Results indicate that two bolts MA2248 and MA2260 are made of pure copper with an average weight percentage of 97.55 % and 98.37 % respectively (Table 6). The composition of these bolts is similar enough to MA2217 to suggest that they are made by the same manufacturer (Table 5). As a first result, this links these bolts from the Taylor material picked up in the 1960 s to the same as those collected from KR7 in 1987. However, MA2234 and MA2252 are quite different. MA2252 has a similar composition to the bolts from KR1 and KR3 (Table 6), indicating that it may have been recovered from KR3 in the 1960 s.

MA2234 has a higher lead content than the typical copper alloys seen from KR1 and KR3. Yet, the lead percentage (average of 5 %) is high enough to be considered an addition. Deliberate addition of lead in copper alloys was for the purpose of lubrication given the friction in on the wearing surfaces of rudder assemblages (McCarthy, 2005: 137). It is therefore likely that MA2234 is possibly associated with a rudder assemblage. If the copper-zinc ratio of MA2234 is evaluated, (Table 6, 67:33), this fastener is more consistent with Muntz or yellow metal and could also have been recovered from KR3.

Five samples from a rudder assemblage, including a pintle (MA2220), spectacle plate (MA2221), an unknown large fitting (possibly rudder base/pivot) (MA2222), a strap with decoration (MA2225) and a bolt (MA2234) were sampled. These artefacts tested positive for copper, zinc, tin, lead and iron. The fittings have less zinc, but more tin and a significant amount of lead. The lead addition to this alloy is clearly visible in their micrographs (Figs. 8–9) and aligns with known addition of lead for lubrication of rudder assemblage surfaces.. These four fittings in this group were manufactured with elements varying from 55.61–70.02 % copper, 10.72–28.51 % zinc, 0.37–4.12 % tin, and 5.32–29.07 % lead. The copper-zinc ratios of the fittings show that they have a much higher copper content, the copper zinc ratio of MA2221, MA2222 and MA2225 varies from 81–86 % copper and 14–19 % zinc. These three fittings are quite similar in composition. The pintle has a copper-zinc ratio of 70/30 in all three tested areas.

Table 6
ESEM results of Taylor Material sampled.

	Cu Wt % average	Zn Wt % average	Pb Wt % average	Cu:Zn average ratio
MA2220 pintle	63.83	27.68	7.42	70:30
MA2221 spectacle plate	63.67	12.8	20.21	84:16
MA2222 rudder pivot	67.79	11.21	16.07	86:14
MA2225 strap	67.79	15.36	12.81	82:18
MA2234 bolt	62.07	30.91	5.00	67:33
MA2248 bolt	97.55	NA (trace)	NA (trace)	NA
MA2252 long/keel bolt	66.56	31.23	NA (trace)	68:32
MA2260 complete through-bolt	98.37	NA (trace)	NA (trace)	NA

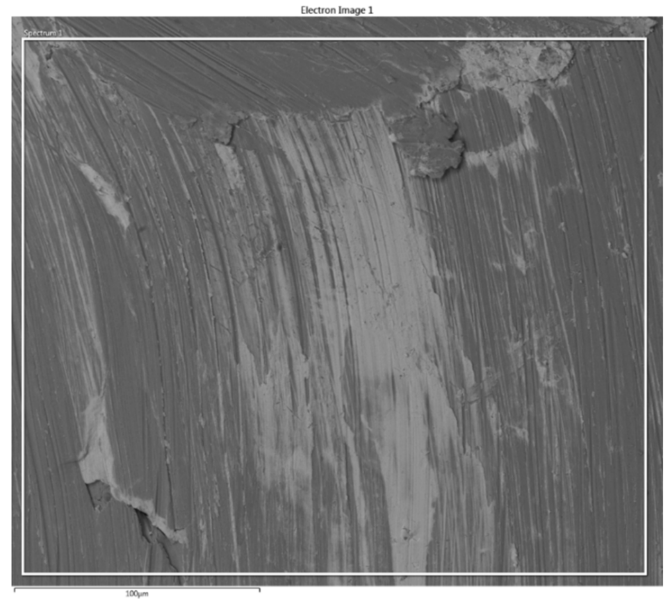

Fig. 8. Micrograph of copper alloy spectacle plate sample, with added lead (white), from the Kenn Reef collection, MA2221, Spectrum 1. Micrograph by W. van Duivenvoorde.

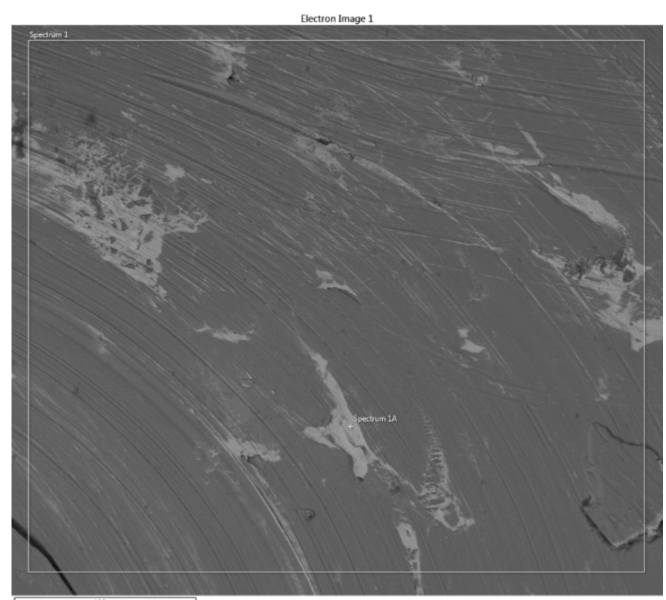

Fig. 9. Micrograph of copper alloy large fitting sample, with added lead (white), from the Kenn Reef collection, MA2222, Spectrum 1. Micrograph by W. van Duivenvoorde.

The rudder assembly artefacts represent percentages similar enough to be from the same manufacturer (pers. comm. Ian MacLeod). Analyses indicate that they are heavily leaded brasses (MacLeod, 1987:283, 285). In addition, comparing the results to other known shipwrecks indicates that the leaded copper alloys, a typical type of alloy for rudder assemblages has similar composition to *Cumberland* built in the UK in 1827. Similar analyses completed by MacLeod (1987:283) gave results of copper (63.0 %), Lead (13.6 %), Zinc (20.5 %), Tin (2.37 %) and Iron (0.86 %). Firmly, placing the construction of the Kenn Reef pintle, spectacle plate, strap and rudder fitting to pre-1840 s. Similar instances of 'pure' copper composition (when excluding lead and zinc additions) such as this are seen in other English-built vessels dated to pre-1840 s (Bennet, 2021; Hunter et al., 2023).

3.5. Summary of results

In terms of the specific results, we can conclude the following. Primarily, that suggested identity of KR1 as the *Doelwijk* (Hosty et al., 2017) is further strengthened by the results of analysing the composition of a copper alloy bolt and fragment of sheathing recovered from the site in 1987 (Delaney and Bately 1987). Analysis of the material recovered from KR3 also indicates that the two sites have material on them from the same date range. In addition, the results also strengthen the argument that KR3 and KR1 are the same site which covers an extensive area across the reef due to extensive site formation processes from the outer to the inner reef edge. To confirm these suggestions, a full site survey should be completed, accurately recording KR1, relocating KR3 and mapping both sites to determine the extent of dispersal.

Results of analysis for KR7 are somewhat interesting. Although the results of the copper alloy sampling indicate a vessel with pure copper fasteners built between 1765 and 1819, the sheathing sample is like yellow metal (Muntz metal) and indicates post 1832. The iron riders found in 2017 most likely suggests a vessel constructed from 1810 onwards and likely much later if it is not of British origin. The disparity in site descriptions between 1987 and 2017 are more concerning. As Delaney and Bately (1987:11) record the site as a scattering of material between several coral outcrops with no substantial structures. Yet, Hosty et al., (2017:85) note only finding a copper alloy bolt, as described by Delaney and Bately (most likely because the described material was all recovered in 1987) but finding extensive and quite substantial additional remains. KR7 should be revisited and accurately surveyed to determine the extent and confirm if the two expeditions are talking about the same site.

Overall, the Taylor material indicates the possibility of another site on the lee side of the reef. If this is true, it may match known information of the construction and final voyage of the American whaler *Lion*. As KR3 was highlighted by Ron Taylor as the most likely original site, yet this could not be relocated in 2017, a thorough survey of that area should be completed in the future to determine the location and extent of KR3, or possibly find a new site. In addition, there are many more copper alloy fasteners in the material donated by the Taylors – ESEM analyses of the full collection may provide more answers.

3.6. Limitations

The authors understood the limitations of the artefact assemblage on Kenn Reef and associated issues of context. A notable limitation is the reliance on the objects to align with the Muntz metal patent from 1832. It is well known extensive use of Muntz's 60:40 copper alloys took approximately another decade to be widely used (Flick, 1975). In addition, differences between countries in terms of shipbuilding practices should be acknowledged. Until further archaeological research is undertaken, and a wider range of comparative analyses are completed, the results give limited insight into the use of chemical composition of copper alloys to date shipwrecks from the nineteenth century. Further limitations arise in the expected refit of ships throughout their lifetime and repairs that introduce varying metals from different sources and different compositions. Ultimately, despite the limitations in confirming results, the analyses provide us with more information and knowledge to pursue.

4. Concluding remarks

Unidentified shipwrecks located on the Great Barrier Reef are not only difficult to access (due to extreme remote locations and environmental conditions) but they also face such turbulent environments that 'good' site preservation is a rare oddity. For that reason, material that remains after centuries on these reef environments is often heavily corroded iron features, anchors, ballast stones and copper alloy fastenings and fittings. A brief outline of the use of copper alloys in shipbuilding given at the start of this article highlights the potential for using and analysing these artefacts to determine much more about these sparse shipwreck sites then was previously possible. The representation of copper alloys in the Queensland Museum State Maritime Archaeology Collection reinforces the high percentage of copper alloys as representative of these shipwrecks. In addition, the minor amount of material required for analysis using an ESEM reduces any ethical implication of destructive analysis and results in valuable compositional data for these sites.

Applying ESEM analyses as a tool for determining the chemical composition of copper alloy artefacts from historic shipwrecks was successful and revealed significant new information. Minimal impact to the artefacts from the sampling method chosen, combined with the minor amount of copper alloy needed, proved to leave nearly indistinguishable marks on museum collection items and maintained the aesthetic and archaeological value of the artefacts. Results of the analyses revealed a wide range of copper alloy compositions and, when compared to historical data, provided valuable insight into these shipwrecks. At this stage, we have proven that compositional analysis of copper alloy artefacts (specific to wooden shipbuilding) using ESEM allows maritime archaeologists to place estimated dates of construction and wrecking on these unidentified sites. This tool provides reliable data that can be referenced to data on known wrecks.

Significantly, when combined with previous archaeological research and historical information, the analyses confirmed the identity of KR1 and KR3 being the same shipwreck site. In addition, our results further cement the preliminary identification of KR1 as *Doelwijk*. While analysing the KR7 material did not provide an exact match to a known wreck, the data also provided insight into the potential construction date of the vessel and allowed us to narrow this down to two possible wrecks. Intriguingly, applying the same analyses to the donated Taylor material proved highly successful in confirming that Ron and Valerie Taylor did collect these artefacts from the vicinity they indicated they were at in the 1960 s. In addition, analysis results reaffirmed the preliminary conclusion that KR7 is likely a vessel built before the 1840 s.

Analysis of the copper alloy sheathing, fasteners and fittings from the Kenn Reef wreck sites provided a range of results. Ultimately, use of ESEM analyses to determine chemical composition of these copper alloys allowed us to estimate dates of construction of these vessels and infer possible dates of wrecking. This study provided key data on Kenn Reef unidentified shipwrecks that was previously unavailable. In addition, it has proved that ESEM is a reliable tool for this approach, given the limitations noted above. Ultimately, the shortcomings of these results lie in the lack of comparative data to draw more solid conclusions and narrow date ranges down.

CRediT authorship contribution statement

Maddy McAllister: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Project administration, Visualization, Writing – original draft, Writing – review & editing. **Wendy van Duivenvoorde:** Formal analysis, Investigation, Methodology, Writing – original draft, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

We have shared the link to the data in previous step. It can be sent on reqeust for reviewing purposes.

Acknowledgements

The authors would like to acknowledge the help from the following people. Ian MacLeod for help with early results and refining comparisons. Myra Stanbury for assistance with other artefact material from the sites. Mack McCarthy for advice on copper alloys fasteners and, Animesh Bashak for his assistance with the analyses and equipment at Adelaide University's, Adelaide Microscopy.

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