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Early vitrification stages identified in prehistoric earthenware ceramics from northern Chile via SEM

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ABSTRACT

Scanning electron microscopy (SEM) has been used to investigate ceramic vitrification stages since the 1970s, however its application has been primarily restricted to stoneware and high-fired ceramics. The purpose of this study was to determine whether stages of vitrification could also be identified in prehistoric low-fired archaeological earthenware ceramics via SEM using northern Chilen samples covering a period c. 2000 to 500 years BP as a case study. This was achieved by visually comparing microstructural changes between original and re-fired ceramic sherds. The microstructural changes identified in this study indicate that the potters who made these vessels achieved early stages of vitrification. This result demonstrates that SEM is a useful technique to investigate the development of firing technology in earthenware manufacture.

1. Introduction

Ceramics are one of the most common materials recovered from archaeological sites. The production of ceramics involves a number of stages including: 1) raw material procurement (clay and tempering material [if required]); 2) processing of raw materials (e.g., the removal of unwanted materials and crushing); 3) manufacturing processes (e.g., mixing of the raw materials, forming the vessel, surface treatment, application of decorative or iconographic features); and 4) firing. The firing process is a key stage to analyse when investigating ceramic technology, as the physical properties of clays change depending on four main factors: the temperature, soak (how long the vessel is exposed to the maximum temperature), the total duration of the firing and the atmosphere.

Ceramics go through different phases during firing. First, organic material (if present) and structural water are burnt off, the initial phase results in a 'stickiness' of the clay particles and partial deconstruction of the mineral structure (sintering), secondly the air spaces collapse, resulting in a liquid phase and, finally, total vitrification occurs (Shepard, 1956:83). When clay is subjected to sufficient firing atmosphere, temperatures, soak and duration it undergoes vitrification (ranging between 800 and 1000 °C depending on the raw materials), which means that the microstructure of the clay body, including clay and tempers, begin to soften and fuse together (Rice, 2005). The firing technology used by ancient potters will have a significant impact on the maximum firing temperature and soak achieved. Kiln firing is able to achieve and sustain higher temperatures (up to 1200 °C) whilst open firing systems reach lower and more varied temperatures and cool more quickly (Chatfield, 2010; Rice, 2005; Tite et al., 1982). In order to determine the maximum temperature to which a sherd was exposed during original firing the clay microstructure must be microscopically analysed to identify the stage of vitrification. Understanding vitrification stages allows archaeologists to infer the type of firing technology employed by potters in the past, which may vary depending on the function and purpose of the vessels.

As noted in Sutton and Arkush (2009:119), total vitrification generally occurs around 1200 °C. However, it should be noted that the temperature required for vitrification to begin is dependent on the clay properties. These differences in properties, as well as the presence of fluxing impurities (such as feldspar which lowers the maturing temperature and promotes vitrification), result in some clays having a lower threshold for the maximum temperature required to begin vitrification (Sutton and Arkush, 2009:119). Further, Shepard (1956:81) and Sutton and Arkush (2009:119) note that variation in the temperature required for vitrification is common in 'low-grade' clays due to clay properties and fluxing impurities (Rice, 2005). For example, clays commonly used by traditional potters worldwide can produce ceramics which reach the initial stages of vitrification at around 800–900 °C (Rice, 2005; Sutton and Arkush, 2009).

One technique that has been employed to investigate the

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vitrification stages of ceramics is scanning electron microscopy (SEM). SEM is capable of producing a highly magnified image (between 2000 and 3000 times magnification) from which the clay microstructure can be observed. For most SEM studies concerned with determining the stage of vitrification, a small fragment is removed from the sherd of interest. This small fragment is then fired to a known temperature (usually between 800 and 900 °C) (see Chatfield, 2007, 2010; Tite, 1992; Tite and Maniatis, 1975; Tite et al., 1982). The original sherd's clay microstructure can then be compared to that of the re-fired fragment. By comparing the two samples, the stage of vitrification for the original sherd can then be identified and used to infer the maximum temperature (as either greater than or less than the maximum temperature used in the re-firing) that it was exposed to during firing (see Chatfield, 2007, 2010; Tite, 1992; Tite and Maniatis, 1975; Tite et al., 1982). The success of this technique has been demonstrated for high-fired ceramics (see Chatfield, 2007, 2010; Tite, 1992; Tite and Maniatis, 1975; Tite et al., 1982), however, the application of this technique on low-fired earthenware is yet to be realised.

2. Background

2.1. The Caleta Vitor Archaeological Complex, northern Chile

The earthenware ceramics used in this study were excavated from the Caleta Vitor Archaeological Complex. This complex is located approximately 30 km south of Arica, on the coast of the Atacama Desert at the mouth of the Vitor or Chaca Valley (see Bird, 1943; Carter, 2016; Bland et al., 2016; Carter, 2016; Latorre et al., 2017; Roberts et al., 2013; Swift et al., 2015) (see Fig. 1). Most of the Atacama region receives < 1 mm of rain per annum (DGA, 2007) and has minimal vegetation making it one of the worlds driest territories, with extremely harsh geo-ecological conditions and marginal terrestrial resources (Grosjean et al., 2007; Marquet et al., 1998, 2002; Ramirez de Bryson et al., 2001; Latorre et al., 2005; Petruzzelli, 2012; Roberts et al., 2013). By exploiting marine resources along the coast and localized sources of fresh water (from Andean summer rainfall), prehistoric people were able to survive on the margins of this arid landscape (Santoro et al., 2005; Roberts et al., 2013; Pestle et al., 2015).

The Vitor Valley is a steep-sided ravine that cuts into the flat desert above to a depth of 400 m (Roberts et al., 2013) (Fig. 2a, b). Caleta Vitor consists of a broad sandy beach bordered to the north and south by cliffs reaching 800 m above sea level (Roberts et al., 2013; Latorre et al., 2017). The archaeological complex, as defined by Carter (2016), includes 'occupation sites, middens, mounds, human remains (including burials and mummy bundles) and various materials such as lithics, ceramics, textiles, fauna/flora remains, and woven matting' (Carter, 2016; Roberts et al., 2013:2361), and spans a large time period, from the Archaic (c. 9000–8000 BP) to the Hispanic Period (c. 476 BP) (Bland et al., 2016; Roberts et al., 2013; Latorre et al., 2017; Swift et al., 2015). For details of changes in material culture (excluding ceramics—see below) over time and their associated cultural phases see Rivera (1991, 2008). For the sake of clarity a regional chronology is summarised in Table 1.

Previous research conducted by Bland et al. (2016) investigated the procurement of raw materials used in the manufacture of ceramics excavated from Caleta Vitor. The objective of the 2016 study was to

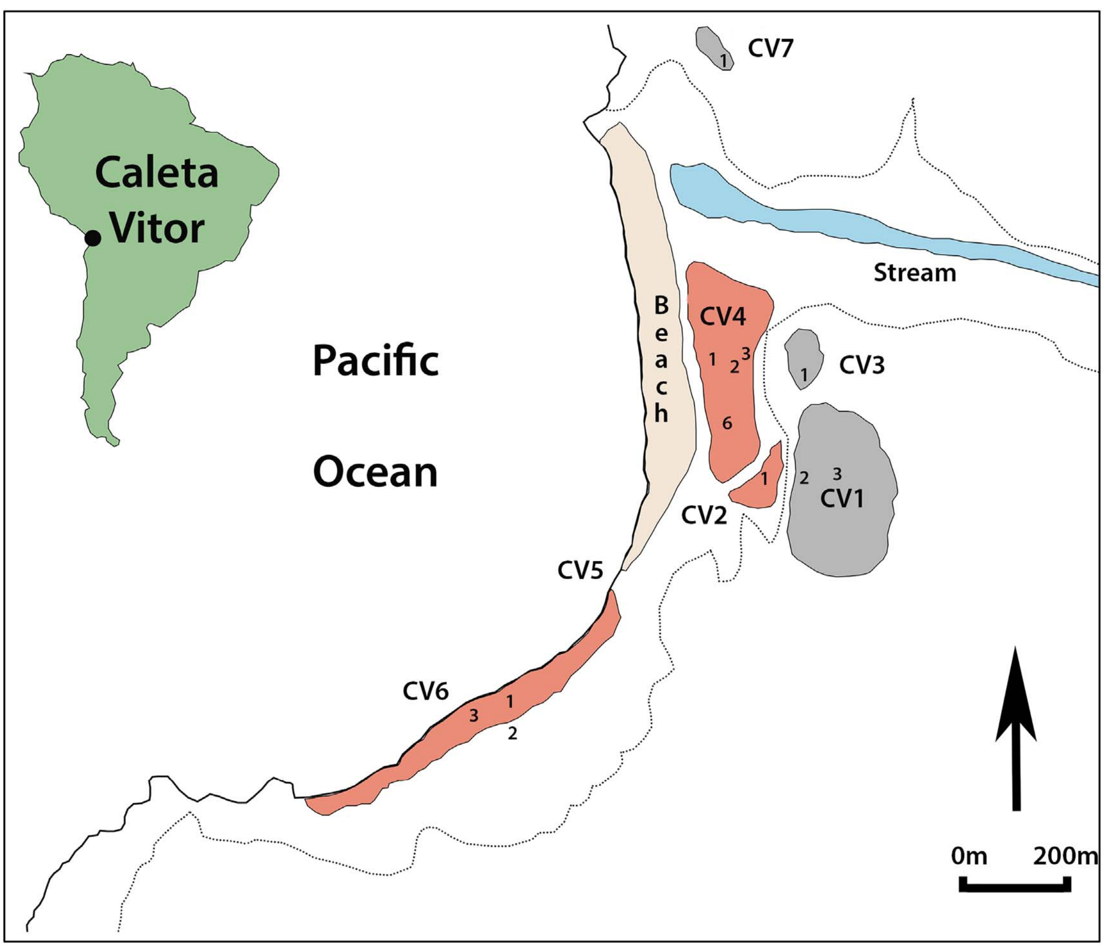

Fig. 1. Location of the Caleta Vitor (CV) Archaeological Complex and location of CV sites and numbered excavated trenches referred to in text (after Carter, 2016). Contour lines indicate the start of the steep-sided ravine. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)