Using WDS to Estimate the Firing Time of Koryŏ Celadons

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Koryŏ period (918-1392 CE) Celadons are widely considered to be aesthetic masterpieces of ancient ceramics [1]. To create similar contemporary examples requires the glaze chemistry and proposed firing conditions (temperature and time). The measurement of the glaze chemistry from a glaze chip ignores the body-glaze interactions during firing [2] that drastically alter the glaze chemistry. Determining the extent of the body-glaze interactions solves both the time problem and the glaze chemistry problem, improving the potential to develop glazes with similar aesthetics to Koryŏ period celadons.

We were provided with several Koryŏ period celadon specimens (shards) and an exquisite 12th century tea bowl from which we extracted a specimen (via core drill) for analysis. (Shard analysis is problematic, as kiln site shards are decidedly reject pieces, so these are ignored in this work.) The core-drilled specimen was mounted in epoxy and polished perpendicular to the body-glaze interface and not etched. Wavelength Dispersive Spectroscopy (WDS) was used to map the body-glaze interface using a JEOL Model JXA-8200, operating at 10 keV, spot diameter of 1 µm, and beam current of 0.03 µA. As shown in Figure 1, the overall glaze thickness is ~390 µm. The original body-glaze interface location is necessary to determine body-glaze interaction depth. Comparison of the Si and Al scans indicated mullite (3Al₂O₃·2SiO₂) in the glaze. Mullite, however, is not a possible reaction product in glaze melting and only precipitates in the body during firing [3]. Since Koryŏ celadons are single-fired products, mullite could only be present in the glaze if it was present in the body prior to firing – previously fired ware was crushed and incorporated into the new body as grog. As the glaze dissolves the body during firing, the mullite particles, which are insoluble in the glaze, remain suspended in the glaze, providing the location of the original body-glaze interface. The body-glaze interaction depth was ~230 µm – 4x greater than typically observed for contemporary glaze systems [4].

To determine the time necessary to obtain a body-glaze interaction depth, a series of glazed samples were prepared and fired for various times at 1250°C and 1300°C [5]. The original body-glaze interface was marked by adding zircon into the body (which is insoluble in the glaze). The results produced three observations (Figure 2): (1) body-glaze interaction depth is log-log with time; (2) single-fire and twice fired systems have similar body-glaze interaction behavior; and (3) similar depth versus time results at 1250°C and 1300°C. Other work on the Koryŏ celadon specimen involving the glass composition in the celadon body indicated a peak firing temperature of 1250°C [6]. The extent of the body-glaze interaction indicated that the tea bowl had been held at peak temperature for ~100 hours (4 days ±1 day).

Such a long firing cycle partially explains the aesthetics of the Koryŏ period glazes and the difficulties in duplicating these glazes. Finally, it is evident from the WDS data, that the iron level in the glaze is identical to that in the body – iron provides the glaze with its characteristic green color, and does not appear to be an initial glaze component but a result of body-glaze interactions.
References:


Figure 1. A back-scatter image and WDS-generated chemical maps of the body-glaze cross-section of a Koryo Dynasty celadon. The presence of mullite, evident in the Si and Al images, indicates the original body-glaze interface and showing the depth of body-glaze interaction during firing.

Figure 2. Glaze penetration depth as a function of time demonstrating a log-log relationship. Three sets of data are presented: 1250°C (single-fire); 1300°C single-fire and twice-fire. Single fire indicates that the glaze is applied to an unfired body and the body and glaze are fired together. Twice-fire indicates that the body is bisqued fired first, then the glaze is applied and the body and glaze are fired a second time. To obtain a penetration depth of 230 μm it was necessary to maintain the peak temperature for ~100 hours (~4 days).