Characterization of Black-glazed Porcelains Using Positron Annihilation Lifetime Spectroscopy

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1. Introduction

The non-destructive nature of positron annihilation lifetime spectroscopy (PALS) has facilitated its implementation in archeological studies [1-2]. PALS is a specialized analytical technique used to study the properties of materials, particularly the structure and defects within them. This technique is applied in various scientific fields, including material science, solid-state physics, and even cultural heritages. PALS can be utilized to analyze archaeological artifacts, historical materials, and artworks to understand their creation and degradation and preservation needs.

The black-glazed porcelain has been popular in various cultures and historical periods. It has a distinctive black-colored glaze applied to its surface. The color and texture of the black-glazed porcelain are contigent upon the firing temperature. This heating process is responsible for converting the clay and glaze into the final porcelain ware.

In this study, we employed PALS to examine the artificial black-glazed porcelains.

2. Materials and Methods

Three black-glazed porcelains were artificially produced at a distinct firing temperature (1,150-1,300°C). The concentrations of iron oxide, ash of oak, calcite, feldspar were 30, 10, 20, and 40 wt.%, respectively (Table 1).

A positron source containing Na-22, enclosed within a titanium window with a 5-µm thickness, was affixed to the artificial black-glazed porcelain sample. PALS system consists of two fast timing plastic crystals (BC-422Q, Saint Gobain Crystals) assembled to two photomultiplier tubes (PMT) (R329-02, Hamamatsu Photonics K.K.), two PMT bases (265A, Ortec), two high-voltage power suppliers (556, Ortec), two constant fractional differential discriminators (CFDDs) (418, Ortec), a nanosecond delay (425A, Ortec), a time-toamplitude converter (TAC) (566, Ortec), and an analogto-digital converter/multi-channel analyzer (927, Ortec) (Fig. 1). The PAL spectra were unfolded into three positron lifetime components using PALSfit3 software. Each spectrum was measured with over two million counts, which required more than 20 hours.

Table 1. Firing temperature and concentrations of the black-glazed porcelain samples.

Sampl Environmen Firing Iron Ash Calcit Feld	lsna
e t $(^{\circ}C)$ oxide Oak e	rspa r
#1 Oxidation 1,300	
#2 Oxidation 1,250 30 10 20 4	0
#3 Oxidation 1,150	



Fig. 1. Setup for positron annihilation lifetime spectroscopy (PALS)

3. Results and Discussion

Table 2 shows the positron lifetimes (τ) and relative intensities (I) for the artificial black-glazed porcelain samples. Fig. 2 presents the positron lifetime spectrum of sample #1. The shortest positron lifetime (τ_1) of the samples ranged from 0.139 to 0.141, which exhibited consistency in terms of their chemical composition. The τ_2 -values were linearly proportional to the firing temperature of the black-glazed porcelain samples from 1,150 to 1,300°C (Fig. 3). The *R*-square value achieved 0.9959. The longest positron lifetime (τ_3), related to ortho-positronium in the free volume of the samples, did not give any correlational information of heating process.

Table 2. Positron lifetimes and relative intensities

Sample			τ ₃ (ns)	<i>I</i> ₁ (%)	I ₂ (%)	I ₃ (%)
#1	0.14	0.41	1.79	55.8	39.4	4.8
#2	0.14	0.40	1.84	55.7	39.8	4.5
#3	0.14	0.38	1.71	55.8	39.6	4.6



Fig. 2. Positron annihilation lifetime (PAL) spectrum of the black-glazed porcelain sample #1.



Fig. 3. Correlation between firing temperature and positron lifetime, τ_2 .

4. Conclusions

In this study, we analyzed the black-glaze porcelain sample fired at different temperatures by PALS. The second shortest positron lifetime, τ_2 , was correlated to the firing temperature. In conclusion, PALS can serve as a non-destructive methods to identify the making process of black-glazed porcelain.

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