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MICROSTRUCTURAL CHARACTERIZATION OF HISTORIC VITRIFIED PAVING BRICK FROM THE STREETS OF SOFIA

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ABSTRACT

In 1907 the city of Sofia, the capital of Bulgaria, made a financial commitment to beautify its urban core – a decision that today is still clearly manifested in the iconic bright yellow bricks that form the wearing surface of the main squares and roads of the downtown region. The inevitable fracturing and crumbling of blocks due to heavy traffic loads has reduced the area covered to half its original extent. Yet a surprising number of these bricks have not needed replacement since their installation over one hundred years ago, which is more than doubling their intended life expectancy. Visual inspection of the bricks also belies the fact that they have been in heavy use since their placement - exposed to pedestrian and vehicular traffic for over a century. Recent research efforts within Bulgaria have focused on the characterization of the historic bricks, and the manufacture of new replacement bricks. Additional microscopic investigations of the original brick are presented here, including results from x-ray diffraction, optical microscopy, micro-hardness testing, and scanning electron microscopy.

KEYWORDS: *vitrified paving brick, microscopy, x-ray diffraction, micro-hardness*

INTRODUCTION

Vitrified paving bricks, fired at higher temperatures and longer durations than typical brick manufacture, result in a highly impermeable and durable product [1]. This is epitomized in the yellow vitrified paving bricks that are a distinct and recognizable feature covering the streets of downtown Sofia, the capital of Bulgaria. They have in many ways transcended their utilitarian purpose; prized for their aesthetic appeal, they are now considered a symbol of Bulgaria's rich national heritage. The nature of their acquisition and installation is steeped in rumours, while their continued impressive mechanical performance as a road wearing surface has puzzled generations of engineers. This paper will summarize what is known of the brick's historical background, while

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investigating their superior mechanical properties through tests performed in the Civil Engineering Department at the University of Toronto.

Historical Context

The prevailing local story is that the bricks arrived as dowry when King Ferdinand was to marry Princess Maria; this idea has perhaps endured for its nostalgic association with famous figures in Bulgarian history. Yet research has unequivocally proved this rumour false, as there are records of the bricks being laid in the city during 1907-1908 [2] – several years after the death of the princess. The more probable explanation is that the Bulgarian king made an order for these bricks at the beginning of the 20^{th} century with the purpose of beautifying Sofia, which led to the eventual procurement and installation of 60,000 m^2 of surface area in the city center [3]. It is believed that the bricks were manufactured in Austria-Hungary, then imported and installed by the Bulgarian company 'Isis'[4].

Mechanical Performance

To this day around one half of the initial paved area is still covered by the original yellow bricks, putting the age of the remaining specimens at over a century - more than doubling the intended lifespan. While some of the pavers have invariably been damaged and removed as a result of heavy traffic volumes through the urban core, it is amazing to see so many still in seemingly perfect condition. Another interesting feature is that the original yellow colour has not been tarnished (Figure 1), which is due to the colour originating from the internal mineralogy of the ceramic rather than an applied surface layer [5].

Figure 1: Untarnished Original Pavers in Sofia, Bulgaria.

Why the bricks still retain such a high quality wearing surface after over a century of heavy usage has long been debated in the scientific community. But it was not until 2013, when the Bulgarian government became interested in rejuvenating certain areas of the city by laying the same type of yellow bricks, that the perfect opportunity and motivation to closer study these historic bricks was presented. The initial objective was to understand the physical properties and what made the original bricks so durable [6], with the eventual goal of recreating exact copies using modern manufacturing techniques [3,4]. While not part of the original investigation, the work done at the University of Toronto confirms these findings with regard to composition, and adds valuable information to the body of work regarding the mechanical properties of the Sofia yellow bricks.

EXPERIMENTAL PROGRAM

To understand why the paving bricks exhibit such excellent durability, samples prepared from pieces of the original yellow pavers were put through a series of tests to establish physical properties and composition. The following section of the paper presents the information gathered from these procedures: x-ray diffraction (XRD), optical microscopy, scanning electron microscopy coupled with x-ray energy dispersive spectroscopy (SEM/EDS), and micro-hardness from depth-sensing indentation (DSI).

XRD

A small sample of brick was crushed using a quartz mortar and pestle to a powder passing the 38 µm sieve, and an XRD pattern collected using a Philips PW1730/PW1050 x-ray diffractometer with Ni-filtered Cu Kα radiation (Figure 2). The diffractometer was fitted with a fixed 1º divergence slit, a Philips PW1752/00 curved graphite monochromator, and a PW1711 detector, and covered a range of 5-65º 2Θ using step increments of 0.02º and collected over a 2 h time period. Peak positions were matched to the feldspar mineral anorthite $CaAl_2Si_2O_8$ (PDF #01-073-0265) and the pyroxene mineral diopside $CaMgSi_2O_6$ (PDF #01-072-1379) using PANalytical X'Pert Highscore Plus software and the ICDD PDF-2 2004 database.

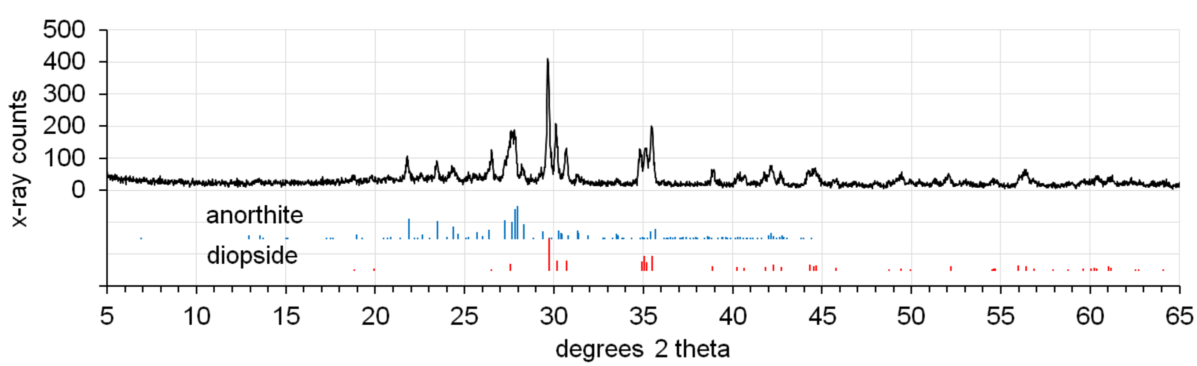

Figure 2: XRD pattern of brick with reference patterns for anorthite and diopside.

Optical Microscopy

A fluorescent epoxy impregnated polished thin section of a brick sample was prepared, and examined with a KOZO XP petrographic microscope equipped for transmitted polarized and fluorescent illumination, with a Luminera INFINITY 2-1R camera. At low magnification, abundant pores can be seen in the fluorescent image, as well as several bright-looking silt-sized quartz SiO_2 grains in the crossed-polars image (Figure 3). At higher magnification, yellow pyroxene crystals are observed, along with clear feldspar crystals (Figure 4).

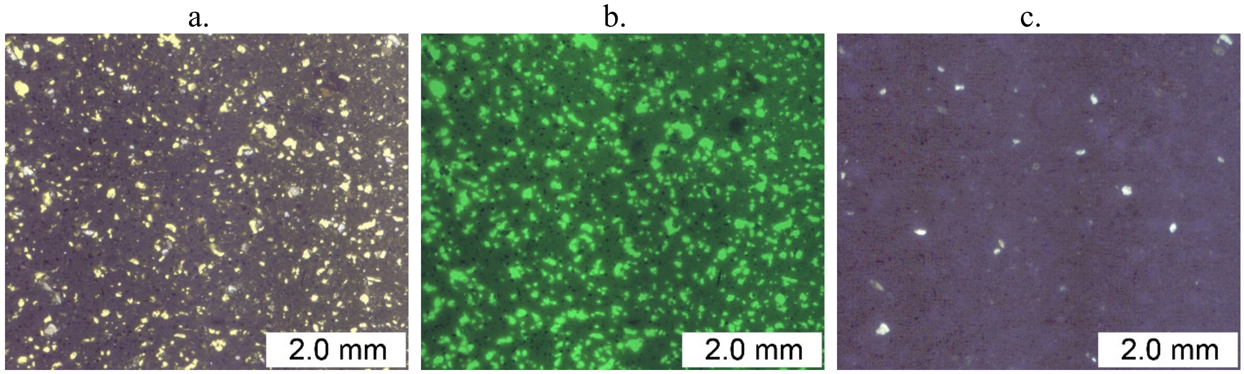

Figure 3: Transmitted plane polarized (a), fluorescent (b), and crossed-polars (c) images from brick thin section, 30 µm thick.

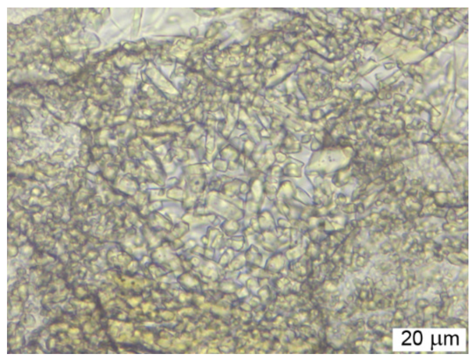

Figure 4: Transmitted plane polarized image from brick thin section, 10 µm thick.

Image analysis software [7] was used to apply a binary threshold [8] to the green band of the RGB image from Figure 2b, to isolate the pores and measure the area. The pores were found to occupy 19 % of the image.

DSI

A Nanovea M1 mechanical tester equipped with a SYNTON-MDP modified Berkovich diamond tip (3 facets with a 65.27º face angle) was used to perform a series of indentations on the polished thin section brick surface. The loading/unloading rate was set at 90 mN/min, to a maximum load of 300 mN, with zero holding time. The contact load was set to 0.1 mN, and the displacement of the tip into the surface was monitored. Since the dimensions of the tip are known, the depth penetrated into the surface can be converted into the projected area in contact with the tip (A). The maximum load (P_{max}) divided by A provides a measurement of micro-hardness (H) expressed in units of GPa (Equation 1) [9].

$$H = \frac{P_{max}}{A} \tag{1}$$

Table 1 lists micro-hardness measurements from the brick, and Table 2 lists micro-hardness measurements from some common minerals [9, 10].

Table 1: Micro-Hardness Measurements from Brick Sample

micro-hardness	measurement						avg.	std. dev.
	1	2	3	4	5	6		
H (GPa)	9.5	11.4	10.9	11.8	11.5	10.4	10.9	0.85

Table 2: DSI Micro-Hardness Measurements for Common Minerals [9, 10]

micro-hardness	mineral				
	corundum	quartz	orthoclase feldspar	diopside	calcite
H (GPA)	29.3 ± 0.4	14.5 ± 0.4	9.1 ± 0.6	8.15	2.2 ± 0.2

SEM/EDS

A thin (200 Å) layer of carbon was evaporated on the surface of the polished thin section, and examined in high-vacuum mode in a JEOL 6610LV SEM at an accelerating voltage of 15 KeV. Back-scattered electron (BSE) images were recorded of the indentations (Figure 5). An OXFORD EDS silicon drift detector was used to produce characteristic Kα x-ray maps of indentation measurement area #5 (Figure 6). For the maps, the beam current was increased to 6 nA, and collected over a period of two hours.

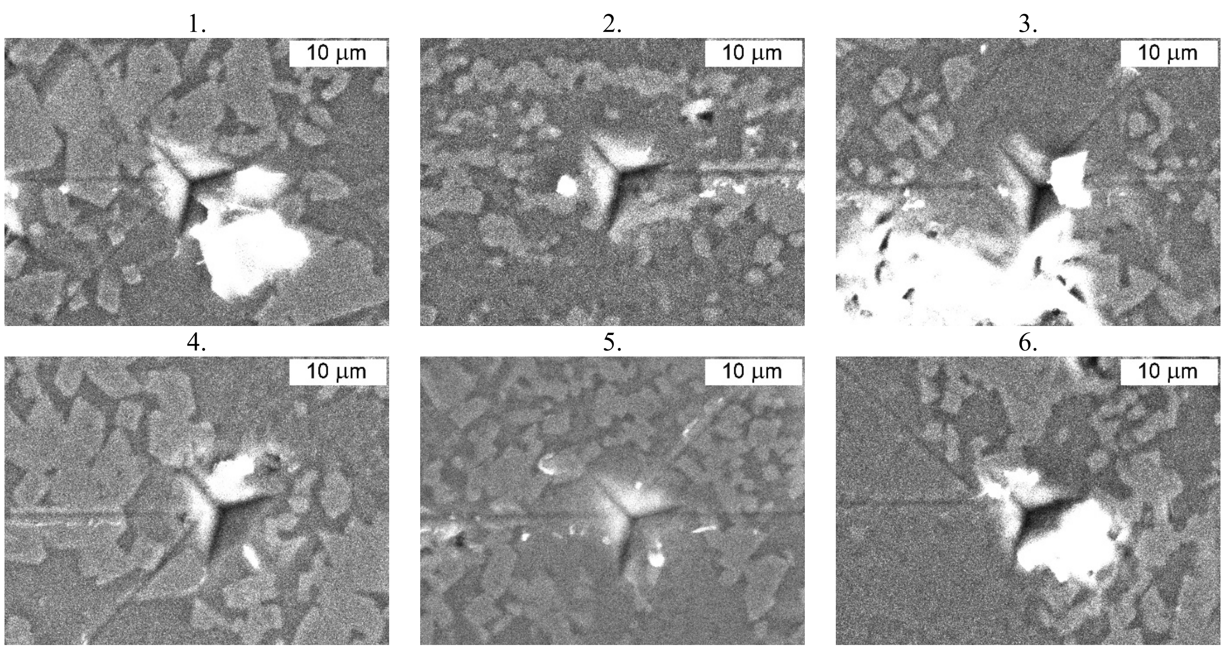

Figure 5: SEM BSE images of indentations #1 through #6.

a. b.

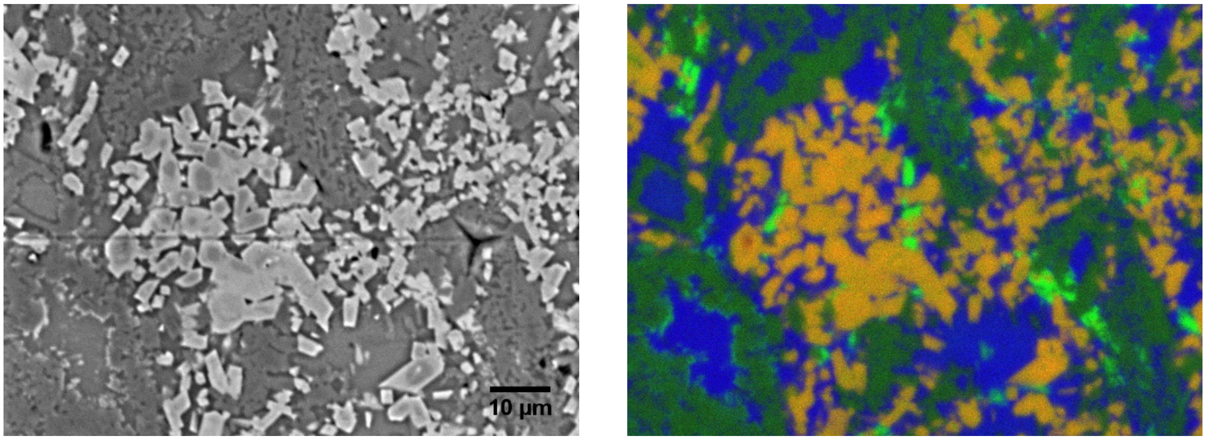

Figure 6: BSE image (a) and RGB color characteristic Kα x-ray map (b) where Mg is assigned to red, Ca is assigned to green, and K is assigned to blue.

DISCUSSION OF RESULTS

The XRD pattern from the powdered brick indicates the presence of both pyroxene and feldspar mineral phases. The optical microscope image of Figure 3 also indicates the presence of remnant quartz grains. Based on the characteristic Kα x-ray map of Figure 6, there appear to be two separate pyroxene phases, the Ca and Mg bearing pyroxene diopside, along with a lesser amount of the Ca end-member wollastonite $Ca_2Si_2O_6$. In Figure 6, diopside appears yellow since it is a combination of the Mg-assigned red band and the Ca-assigned green band, and wollastonite appears bright green because it is Ca-rich (green band) and has little or no Mg (red band) or K (blue band). Similarly, from Figure 6, there appear to be two feldspar phases present, both the Ca end-member anorthite, and a K-bearing orthoclase $KAlSi_3O_8$. In Figure 6, orthoclase appears blue since it is K-rich (blue band), and anorthite appears dark green since it is Ca-rich (green band) with little or no Mg (red band) or K (blue band). The anorthite is dark green as opposed to the bright green wollastonite, since it has less Ca compared to wollastonite (14.4 vs. 34.5 wt. % Ca). Given this combination of phases, the parent material appears to have been a silty carbonate clay material, where the carbonate fraction consisted of both calcite, $CaCO_3$ and dolomite $CaMg(CO_3)_2$, and an illite $KAl_2(Si_3Al)O_{10}(OH)_2$ rich clay [11]. From the fluorescent image of Figure 3 the porosity appears relatively high, but the pores are isolated and not interconnected. The scale of the micro-hardness tests from Figure 5 precluded obtaining results from individual phases. As such, the micro-hardness values from Table 1 reflect the contribution of multiple phases. Compared to the results reported in the literature, the brick had micro-hardness values slightly higher than would be expected for a pyroxene and feldspar combination.

CONCLUSION

While Sofia's yellow road paver bricks were initially intended as simply an aesthetic improvement to the downtown core, it is undoubtedly their longevity and excellent performance that has cemented their legacy. After over a century of heavy use their appearance is untarnished and they continue to provide an adequate wearing surface in all weather conditions. From the work performed at the University of Toronto and discussed in this paper, the conclusion is that the pavers' superior durability can be attributed to three factors: mineral uniformity throughout the brick, high impermeability due to isolated and non-interconnected pores, and reduced abrasion due to high material hardness.

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