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The contribution of technical ceramic to iron smelting production at Sungai Batu, Bujang Valley, Kedah

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Abstract: Iron smelting sites at Sungai Batu, Bujang Valley are dated from 788 - 537 BCE (Beta-516413) until 15th Century CE. The evidence of 17 sites found with the abundance of iron slags and other artefacts clearly show that a large scale of iron-smelting industry existed in Bujang Valley. The term 'technical ceramics' refers generally to any ceramics used in metallurgical or other high-temperature operations. The technical ceramic found in archaeological sites can be classified as furnace, tuyere and brick. They were made from clay and play different roles for iron smelting process. This article will discuss the significant of technical ceramic regarding the impact of iron production. The methods consist of collecting sample from archaeological site, augering sample and scientific analysis such as X-ray fluorescence (XRF), X-ray diffraction (XRD) and scanning electron microscopes-energy dispersive X-ray (SEM-EDX) analysis. The following discussion will further explain the selection of material and particular shapes in enhancing the iron smelting process at Sungai Batu. The result demonstrate that the technical ceramics were made from the locally available with some sand as temper.

Abstrak: Tapak peleburan besi di Sungai Batu, Lembah Bujang berusia dari 535 Sebelum Masihi hingga kurun ke-15 Masihi. Bukti 17 tapak yang dijumpai dengan sisa besi dan artifak lain jelas menunjukkan bahawa industri besi berskala besar wujud di Lembah Bujang, Istilah 'seramik teknikal' secara umum merujuk kepada seramik yang digunakan dalam operasi mertalugi atau operasi lain yang menggunakan suhu tinggi. Seramik teknikal yang terdapat di tapak arkeologi dapat diklasifikasikan sebagai relau, tuyere dan bata. Artifak ini diperbuat daripada tanah liat dan mempunyai fungsi yang berbeza dalam proses peleburan besi. Artikel ini akan membincangkan kepentingan seramik teknikal terhadap penghasilan besi. Kaedah analisis melibatkan pengumpulan sampel dari lapangan, penggerudian cetek dan analisis saintifik secara pendarfluor sinar-X (XRF), pembelauan sinar-X (XRD) dan mikroskop electron-sinar X penyebaran tenaga (SEM-EDX). Perbincangan juga akan menjelaskan lebih lanjut pemilihan bahan dan bentuk tertentu dalam meningkatkan proses peleburan besi di Sungai Batu. Hasil kajian menunjukkan bahawa seramik teknikal diperbuat dari kawasan tempatan dengan sedikit pasir sebagai bajaan.

Keywords: Technical ceramics, iron smelting, Bujang Valley, tuyere, furnace

INTRODUCTION

Sungai Batu site was first discovered during a survey in 2007 (Mokhtar *et al.*, 2011). A survey of Sungai Batu estates, an area of four-square km, revealed a total of ninety-seven mounds, potential protohistoric sites of the Bujang Valley. Excavations from 2009 until recent have uncovered monument site function as a ritual site, 11 structures of jetty, 16 administrations building and 17 iron smelting sites.

Iron smelting sites at Sungai Batu, Bujang Valley are dated from 788 - 537 BCE (Beta-516413) until 15th Century CE (Naizatul & Mokhtar, 2018 & 2019). The iron smelting site reveals iron slag, iron ore, technical ceramics and deposits remains such as ash and charcoal. Iron are produced using direct method which is also refered as bloomery process. In simplified terms, this involved reducing part of the iron oxides of the charge to metal particles that coalesced to form a bloom, consisting of iron uniformly mixed with slag that required hammering to be wrought iron (Rostoker & Bronson, 1990).

The technical ceramics are one of the dominant discarded material in smelting process besides iron slag. The term 'technical ceramics' refers generally to any ceramics used in metallurgical or other high-temperature operations (Martinón-Torres & Rehren, 2014). Technical ceramics are essential tools for almost all metallurgical processes and were routinely exposed to a variety of conditions that they had to cope with (Martinón-Torres & Rehren, 2014). The technical ceramic found in archaeological sites can be classified as furnace, tuyere and brick. They were made from clay and have a different role to iron smelting process.

This paper explores the role technical ceramics play in various aspects of the Sungai Batu iron smelting site and the selection of the best material for technical ceramic. The main focus will be with the furnace and tuyères and the source of the raw material.

METHODOLOGY

The method consists of collecting sample from archaeological site and augering sample for scientific analysis such as X-ray fluorescence (XRF), X-ray diffraction (XRD) and scanning electron microscopes-energy dispersive X-ray (SEM-EDX) analysis. The sampling process involved tuyere and furnace pieces from SB2A, SB2C, SB2F, SB2H, SB1G and SB1ZY site (Table 1).

The augering sampling using AMS basic soil auger kit covered the area near the ancient river of Sungai Batu. The location of the drilling was randomly selected in the area near the ancient river by referring to the result done in 2009 (Nor Khairunnisa, 2009). A total of six clay samples were collected from three different localities (Log 1, Log 2, Log 3) (Figure 1).

CLAY SOURCE

Sungai Batu Complex area was situated between Gunung Jerai (north) and Sungai Muda (south) within

Table 1: Sample type and location.

Type	Sample No.	Location	Sample No.	Location
Clay	C1	Log 1, AD:105cm	C4	Log 2, AD:150cm
	C2	Log 1, AD:130cm	C5	Log 3, AD:120cm
	C3	Log 2, AD:100cm	C6	Log 3, AD:158cm
Furnace	D1	SB2A (Q10)	D6	SB2F (J9)
	D2	SB2A (O10)	D7	SB2F (K9)
	D3	SB2C (K16)	D8	SB1ZY (H5)
	D4	SB1G (J11)	D9	SB1ZY (J6)
	D5	SB1G (J12)	D10	SB2H (N1)
Tuyere	T1	SB2A (P15)	T7	SB2F (T4)
	T2	SB2A (R8)	T8	SB2F (G8)
	T3	SB2C (M25)	T9	SB1ZY (G8)
	T4	SB2C (L28)	T10	SB1ZY (J12)
	T5	SB1G (F14)	T11	SB2H (J12)
	T6	SB1G (T10)	T12	SB2H (S5)

*AD: Actual Depth; ( ): trench

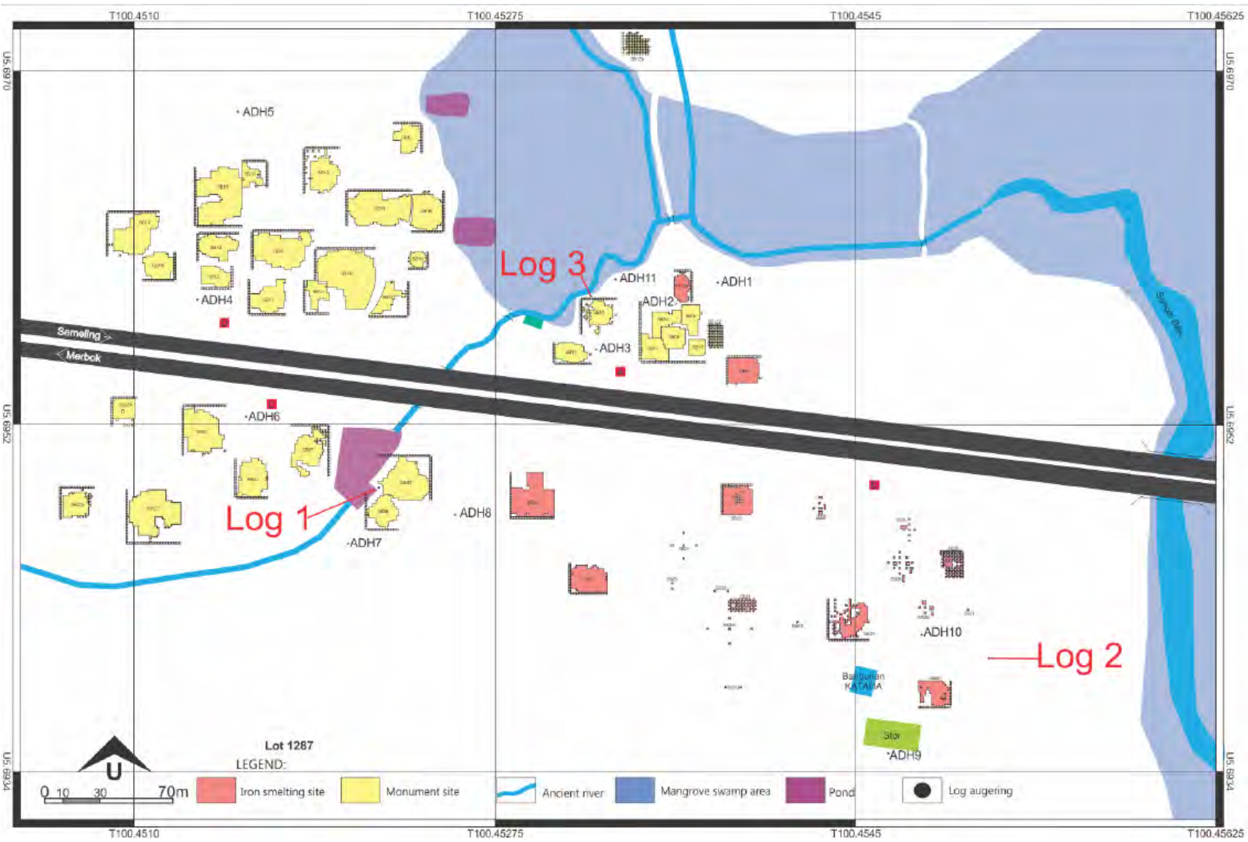

Figure 1: The location of iron smelting site and augering samples.

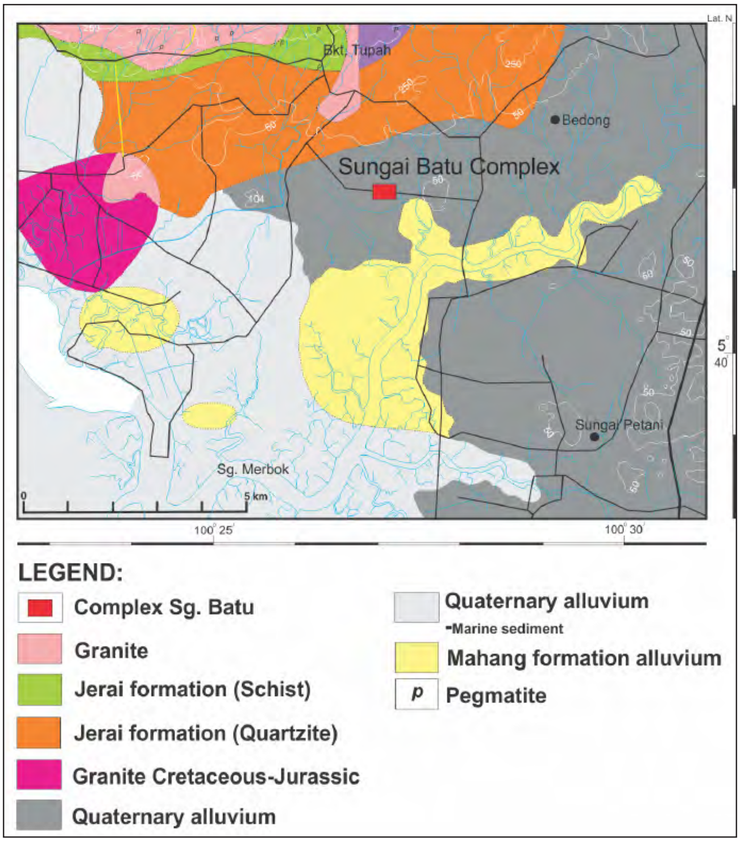

Figure 2: Geological map of the study area.

Kuala Muda, Kedah. The study area is an alluvial land with abundant iron ore distribution in the southern part of Kampung Merbok (Bean, 1969; Bradford, 1972) (Figure 2). The soil types of the area are sandy clay covered with fine sand. There are few small rivers and swamp area at the eastern part of the area.

FURNACE

Furnaces are typically immobile structures that contain the charge, such as charcoal and minerals, and are used for the smelting of ore to metal iron (Martinón-Torres & Rehren, 2014). Furnace found at iron smelting site are in fragmented pieces and the remains of the furnace can be seen at the base (Figure 3). They are in small pieces, irregular shapes and were made from clay. The width of the furnace remains are estimated around 100 cm and there were two to three furnace remains found in each site.

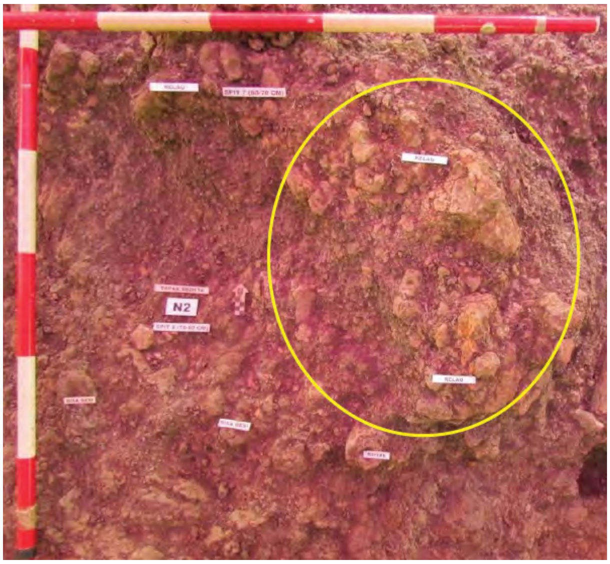

Figure 3: Remaining furnace.

TUYERE

Tuyere is one of the important tools in ancient iron smelting. Its main function is to control and balance the combustion temperature during the smelting activity. The hole in the center of the tuyere allows the air supply and oxygen that are needed to increase the temperature in the furnace (Figures 4 & 5). Most of the tuyeres found at the iron smelting site are in broken shapes. They are classified as a waste product and more than hundred thousand fragmented pieces have been excavated so far. The recorded number of tuyere represent only artefacts that have been excavated, yet there are more still uncounted which are buried in the mound.

Figure 4: Tuyere.

Figure 5: Tuyere from the sideview.

A 'complete' tuyere means that it ranges from a rear end where the bellow was inserted, to a vitrified nozzle (inside the furnace) (Veldhuijzen, 2005). A total of 101 complete tuyeres have been taken out from the field. No definite conclusions can be drawn about the original full length of the tuyères but the longest tuyere found measured about 27.1 cm (length) and 11.2 cm (width). The inner diameter of the tuyere (inner hole) is approximately 2.1 cm to 3.6 cm (Figure 6).

This analysis of complete tuyeres show that they were made with standardization in shape and size. The standardization of tuyere production helps to increase maximum efficiency for the smelting operations. The air blown in from the tuyeres does affect the reduction process in the furnace (Friede *et al.*, 1984). The features of tuyeres also suggested that a hand technique was used to form the objects and have been molded around a stick to make the hole (Venunan, 2011).

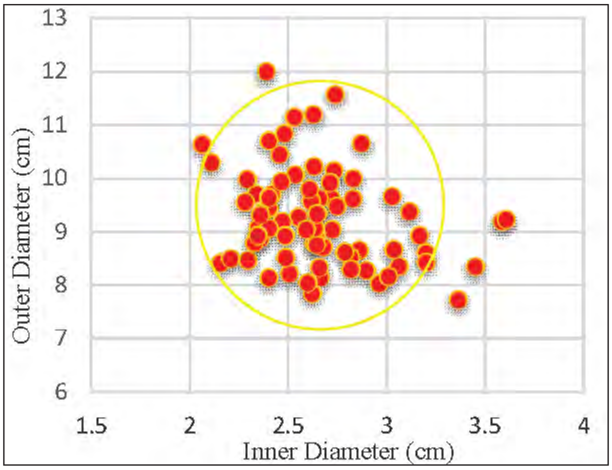

Figure 6: Diagram of diameter samples of tuyere for inner and outer diameter.

RESULT

The XRD results show that the minerals found in the furnace and tuyeres are quartz, dickite, rutile, mullite, cristobalite, tridymite, montmorillonite, diaspore, muscovite, microcline, fayalite, anorthite, hematite, dolomite and magnemite (Table 2). There are many high temperature phase minerals (e.g., mullite, cristobalite and tridymite) which transformed from kaolinite and quartz, found in the furnace and tuyere samples. Quartz, dickite, rutile, diaspore and muscovite can be found in the clay samples. Kaolinite and quartz undergo some significant changes in its structure when heated to higher temperatures. Mineral transformation series was made over the range: kaolinite-mullite 950-1500°C, quartz-tridymite 866.85-1469.85°C and cristobalite 1469.85-1679.85°C. The quartz was added as a temper, which would have improved the toughness and thermal shock resistance of these technical ceramics (Venunan, 2011).

The XRF analyses present a ceramic matrix with approximately 65-75 wt% SiO_2 and roughly 10-17 wt% Al_2O_3 (Table 3). The higher SiO_2 and Al_2O_3 content in technical ceramics can be explained by the nature of technical ceramics which need to be more refractory, and silica in this case influenced the level of technical ceramics to be tolerant enough to survive the operation (Venunan, 2011). The percentage ratio of alumina to silica for 10 furnace samples is between 1: 4.1 to 1: 7.5 (Table 3). On the other hand, the percentage ratio of alumina to silica in the manufacture of tuyere is between 1: 3.9 to 1: 4.6. Comparison of alumina ratio to silica between furnace and tuyere shows that the use of silica (sand) in the furnace manufacturing was more than in the tuyere production. This is due to the need for the furnace structure to last longer and the presence of quartz as a temper can increase the resistance to high temperatures (Venunan, 2011). The temperature at the bottom of the furnace is higher compared to at the other parts of the furnace, where the temperature is about 1200°C (Thiele, 2010).

Observation under the optical microscope show that quartz grains are predominant in the fabric of technical

Table 2: XRD results.

Sample no.	Mineral																
	Q	Kao	Dic	Rut	Kris	Mul	Tri	Mont	Dia	Mus	Illite	Micro	Fay	Anor	He	Do	Magn	Mag
C1	•	•	-	-	-	-	-	-	-	•	-	-	-	-	-	-	-	-
C2	•	•	•	•	-	-	-	-	-	•	-	-	-	-	-	-	-	-
C3	•	•	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
C4	•	•	-	-	-	-	-	-	•	-	-	-	-	-	-	-	•	-
C5	•	•	-	-	-	-	-	-	•	-	-	-	-	-	-	-	-	-
C6	•	•	-	-	-	-	-	-	•	•	-	-	-	•	-	-	-	-
D1	•	-	-	-	-	-	-	•	•	-	-	-	-	-	-	-	-	-
D2	•	-	-	-	-	-	-	•	-	-	-	-	-	-	-	-	-	-
D3	•	-	-	-	-	-	-	-	-	•	-	-	•	-	-	-	-	-
D4	•	-	-	-	-	-	-	•	-	-	-	-	-	-	•	-	-	-
D5	•	-	-	-	-	-	-	•	-	-	-	-	-	-	•	-	-	-
D6	•	-	-	-	-	-	-	•	-	-	-	-	-	•	-	-	-	-
D7	•	-	-	-	-	-	-	-	-	-	•	-	-	-	-	-	-	-
D8	•	-	-	-	-	-	-	•	-	-	-	-	-	-	-	-	-	-
D9	•	-	-	-	-	-	-	•	-	-	-	-	-	-	-	-	-	-
D10	•	-	-	-	-	-	-	•	-	•	-	-	-	-	-	-	-	-
T1	•	-	-	-	•	•	-	-	-	-	-	-	-	-	-	-	-	-
T2	•	-	-	-	-	-	-	-	-	•	-	-	-	-	-	-	-	•
T3	•	-	-	-	-	•	-	-	-	-	-	-	-	-	-	•	-	-
T4	•	-	-	-	•	•	-	-	-	-	-	-	-	-	-	-	-	-
T5	•	-	-	-	-	-	-	•	-	-	•	-	-	-	-	-	-	-
T6	•	-	-	-	•	•	-	-	-	-	•	-	-	-	-	-	-	-
T7	•	-	-	-	•	•	-	-	-	-	-	-	-	-	-	-	-	-
T8	•	-	-	-	-	-	-	•	-	•	-	-	-	-	-	-	-	-
T9	•	-	-	-	•	•	-	-	-	-	-	-	-	-	-	-	-	-
T10	•	-	-	-	-	-	-	•	-	-	-	•	-	-	•	-	-	-
T11	•	-	-	-	•	•	•	-	-	-	-	-	-	-	-	-	-	-
T12	•	-	-	-	-	-	-	•	•	-	-	-	-	-	-	-	-	-

Notes: Q: quartz, Kao: kaolinite; Dic: dickite; Rut: rutile; Mul: mullite; Cris: cristobalite; Tri: tridymite, Mont: montmorillonite; Dia: diaspore; Mus: muscovite; Micro: microcline; Fay: fayalite; Anor: anorthite; He: hematite; Do: dolomite; Magn: magnetite; Mag: magnemite

ceramics. The SEM analysis revealed that all technical ceramic samples are exposed to high temperature, as shown by the presence of cracked quartz and dissolved clay matrix (Figures 7 and 8). The cracked and dissolved quartz indicate that a fluxing agent for lowering the temperature to dissolve quartz were added in the smelting operation. The molten technical ceramic contributes actively to the formation of the slag (Veldhuijzen, 2005). Therefore, this will facilitate the production of the metal.

In order to assess the clays that was used to produce the technical ceramics, the chemical composition of clay samples was compared with the technical ceramics (Figure 9). The comparison of bulk compositions also demonstrates the similarity between the technical ceramic and the clay samples from three localities above 100 cm to 130 cm. The results clearly show that the raw materials were collected near the ancient river of Sungai Batu, with the estimated depth 100 cm to 130 cm.

CONCLUSION

The technical ceramics found at Sungai Batu iron smelting site play a large and crucial role in the iron

Table 3: XRF result.

Sample no.	Chemical composition (% Weight)										
	SiO_2	TiO_2	Al_2O_3	Fe_2O_3(t)	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	Cl	SO_3
C1	69.72	0.94	16.09	1.04	0.01	0.20	0.06	0.10	0.48	0.08	-	-
C2	59.67	0.91	24.30	1.29	0.01	0.17	0.05	0.10	0.43	0.03	-	-
C3	72.28	0.74	14.70	1.54	0.01	0.24	0.06	0.09	0.51	0.03	-	-
C4	64.67	0.83	20.87	1.22	0.02	0.26	0.06	0.13	0.73	0.03	-	-
C5	65.53	0.66	15.32	2.07	0.01	0.31	0.12	0.39	0.73	0.04	-	3.31
C6	53.02	1.04	29.00	1.38	0.02	0.28	0.06	0.27	1.43	0.05	-	-
D1	75.74	0.54	11.91	2.74	0.17	0.33	0.46	0.15	0.51	0.04	-	-
D2	68.26	0.69	15.12	3.04	0.03	0.31	0.45	0.35	0.41	0.06	-	-
D3	63.66	0.62	15.54	2.57	0.05	1.06	0.96	1.27	0.44	0.10	3.74	-
D4	65.20	0.60	10.97	12.7	0.16	0.27	0.38	0.39	0.36	0.13	-	-
D5	62.94	0.64	11.71	13.86	0.13	0.18	0.21	0.06	0.38	0.12	-	-
D6	77.16	0.52	10.33	2.97	0.03	0.17	0.11	0.11	0.32	0.05	-	-
D7	71.97	0.92	15.40	4.19	0.02	0.32	0.06	0.03	0.25	0.05	-	-
D8	74.90	0.70	10.61	3.09	0.01	0.27	0.05	0.04	0.52	0.04	-	-
D9	69.11	0.77	12.38	6.49	0.01	0.24	0.05	0.04	0.66	0.09	-	-
D10	65.92	0.74	13.78	4.80	0.12	0.50	2.14	0.08	0.36	0.12	-	-
T1	74.03	0.62	17.03	1.92	0.02	0.24	0.07	0.18	0.70	0.04	-	-
T2	72.86	0.57	12.64	2.15	0.07	0.18	0.24	0.12	0.32	0.07	-	-
T3	70.52	0.83	17.16	5.21	0.01	0.33	0.04	0.13	0.34	0.06	-	-
T4	72.16	0.75	14.66	4.98	0.02	0.55	0.64	0.05	0.86	0.07	-	-
T5	72.16	0.74	13.64	3.07	0.01	0.11	0.08	0.05	0.50	0.07	-	-
T6	71.94	0.77	16.56	3.93	0.02	0.30	0.04	0.05	0.35	0.09	-	-
T7	73.28	0.77	16.42	2.98	0.02	0.23	0.05	0.09	0.24	0.08	-	-
T8	67.70	0.85	19.23	2.17	0.01	0.17	0.04	0.12	0.33	0.07	-	-
T9	72.34	0.84	18.35	3.41	0.01	0.28	0.04	0.06	0.25	0.04	-	-
T10	68.47	0.78	15.27	4.39	0.01	0.26	0.04	0.11	0.73	0.04	-	-
T11	68.80	0.97	21.36	2.43	0.01	0.35	0.05	0.24	0.51	0.07	-	-
T12	72.69	0.66	13.96	2.11	0.01	0.15	0.04	0.11	0.36	0.07	-	-

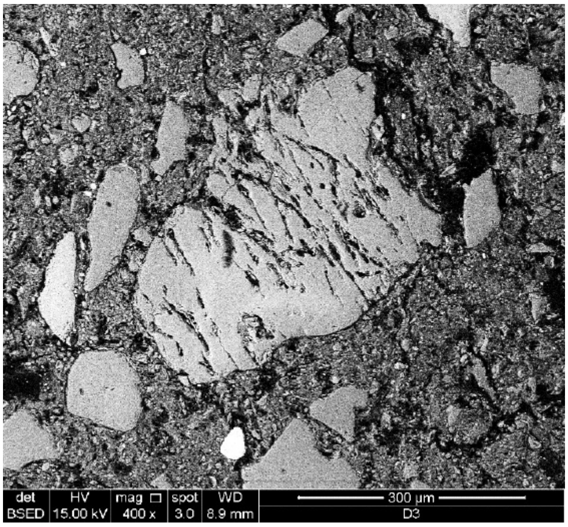

Figure 7: The quartz grains in furnace sample show cracks caused by the heat.

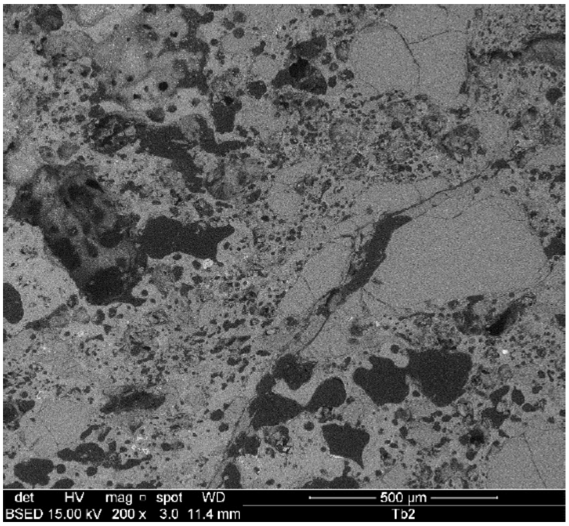

Figure 8: The vitrified region of tuyere show cracked quartz grains and dissolved clay matrix.

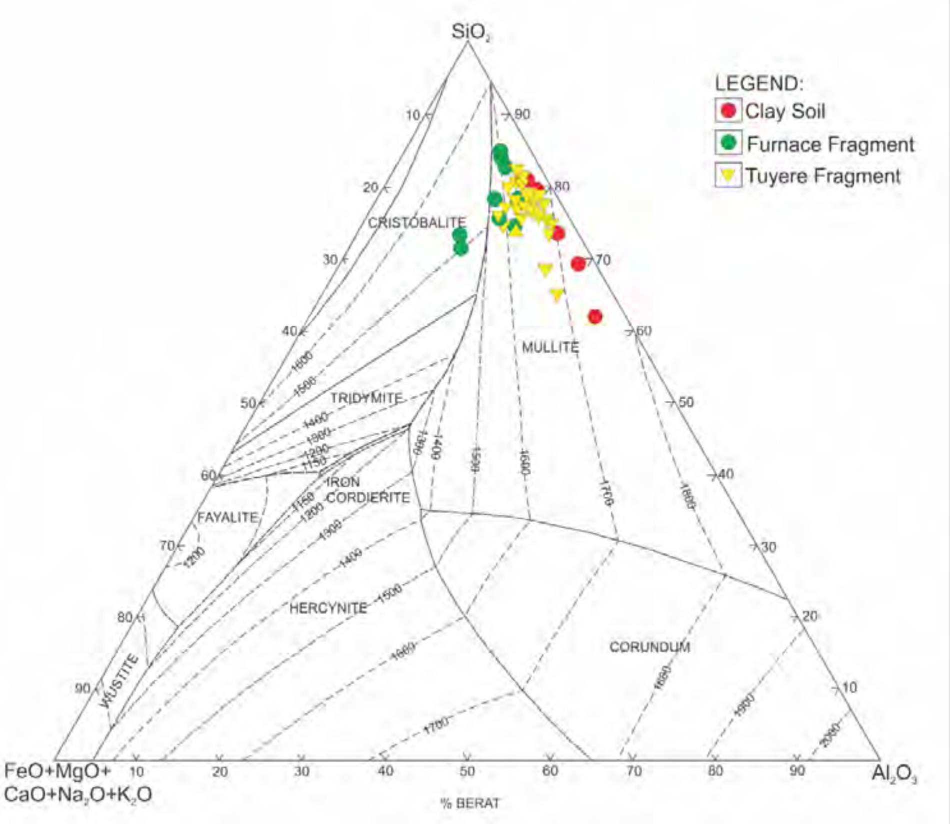

Figure 9: Ternary diagram showing bulk compositions of two technical ceramics compared with clay sample.

smelting operations. From the physical appearance of the tuyeres alone, it can be concluded that a shaft furnace as well as bellows were used. The high number and standardized size and shape of the tuyeres found at Sungai Batu shows that the industry was operating a big scale production. The typical cylinder shape of the tuyeres may tie in with local or regional metallurgical traditions or technological choice. From the combination of these physical and chemical data, it can be concluded that the Sungai Batu smelters purposefully applied tuyeres to facilitate the production of iron. The functions of furnace were not only focused on the temperature they could be sustained but also the selection of best raw material for increasing the production of iron. The site which is located in the direct vicinity of natural resources required in iron production such as water, ore and clay made the Sungai Batu Complex as a strategic location for iron industry.

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