

Design and Development of Low Scale, High Temperature, Hybrid Furnace for the Extraction of Metallurgical Grade Silicon from Raw Mineral Quartz

Engineer Zaib Ullah Khan¹, Prof. Dr. Nasim A. Khan², Prof. Dr. S. Jawid Askari³, Dr Imran Amin⁴

¹ Research Scholar in Energy Engineering, Faculty of Engineering Science & Technology, Hamdard University, Karachi 74600, Pakistan, rajazaibkhan@yahoo.com, rajazaibkahn@gmail.com

² Prof. in Energy Engineering, Senior Executive Director, Osmani & Company (Consultant Engineers-Architects-Planners), Karachi 74600, Pakistan. Ex Vice Chancellor, Hamdard University, Karachi, Pakistan.

³ Prof and Head of the Department, Materials Engineering Department, Dawood College of Engineering and Technology, Karachi

74000, Pakistan. Ex HOD Manufacturing Engineering Department, National University of Science and Technology, Karachi, Pakistan. Ex Researcher, Department of High Tech Thin Film, School of Material Science and Engineering, University of Science and Technology Beijing, Beijing 100083, P R of China.

⁴ Head-Centre for renewable energy research, Shaheed Zulfiqar Ali Bhutto Institute of Science and Technology, Karachi, 74000, Pakistan

Abstract: High temperature furnaces are the most suitable furnaces with the capabilities necessary to proceed further in the research area of obtaining cheap and high quality Metallurgical Grade Silicon (MGS). The Silicon Dioxide (SiO₂) / Silicate generally known as quartz is a hard crystalline mineral stone abundantly available in large quantities all over the world in a variety of appearances comprising about 26 % of the earth layer with other elements (mostly oxygen). In Pakistan this mineral is available excessively in Jhangshahi, Sindh and other parts of the country, but is not being used in extracting MGS due to non-availability of high temperature specialized furnaces. However this mineral is used in construction industries and in different steel manufacturing industries as slag. The element silicon which is a known semiconductor (in which electrons are relatively tightly bound until some change in their environment causes them to flow freely) can be obtained through a series of purification processes of MGS (which can be extracted from this mineral quartz through specially designed low scale, high temperature hybrid furnace). The melting of quartz requires a temperature of around 2000°C, with a control environment to obtain MGS with a suitable silicon purity level of around 90 – 95%. During this research work a hybrid furnace with low scale is designed and developed that can attain temperature range of around 2000°C in order to achieve proper and thorough melting of specially prepared raw mineral quartz. The capacity of this furnace is anticipated to be 3-4 Kg of charge initially, which can be further enhanced with both gas as well as electrical firing option. The tests showed that a time of 60-90 minutes is required to heat up the furnace completely in order to attain the required temperature. The time required to melt the first charge of 1 kg was approximately 2 hours at the melting rate of 8.30 gram / minute with average electrode consumption of 0.0467cm / minute (when fired electrically) while average gas consumption of 90 ccf (when fired with gas option). The average MGS production during this research work is anticipated to be 300 grams approximately from 1 kg of raw mineral quartz.

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1. Introduction

Designing and development of small scale, high temperature, hybrid furnace (both electrical and gas fired) for the extraction of metallurgical grade silicon at a high temperature of around 2000°C is an essential requirement for this research work. The mineral quartz (abundantly available in Jhangshahi, Sindh and other parts of Pakistan) is a hard crystalline, second

largest and the most common element on the earth's surface after oxygen constituting more than 25 % of the earth crust at an average. It is available in many shapes and in combination with other elements, the most common is with oxygen thus known as silicon dioxide (SiO₂). According to the survey only Jhangshahi, Sindh area of Pakistan is having a proven reserve of more than 40 million metric tons of mineral

quartz [1, 2]. As per the analysis report of this mineral from Pakistan Council of Scientific and Industrial Research (PCSIR) laboratory complex, Karachi, Pakistan, this mineral quartz is composed of approximately 96% of silicon dioxide (SiO_2) [3]. Because of its numerous uses and especially acquiring of metallurgical grade silicon, this mineral is considered as a very important element [4].

The important semiconductor characteristic of silicon has revolutionized electronic industry and motivates scientists and engineers to work on this element in order to achieve advancement in electronic industry. The most famous and highly technical industrial area in California, USA which is known as "Silicon Valley" is also named for the same element [5].

During this research work a high temperature, small scale, hybrid furnace is developed which can achieve requisite temperature conditions for melting of mineral quartz (prepared chemically). Since this mineral quartz contain 96 % of Silicon dioxide, so extraction of metallurgical grade silicon from this mineral quartz is encouraged. Presently large scale electrical arc furnaces are available with high temperature of more than 2000 °C in Pakistan steel mills, Karachi and people steel mills, Karachi but they are very large and complicated in design and so expensive that it cannot be used for this type of research and experimental work.

The high temperature, small scale furnaces have several advantages which are helpful for metallurgical research including handy control over temperature and heat, accurate melt analysis, certain smelting and refining arrangements and great thermal efficiency [6]. Much information relating to the design and fabrication of small scale, high temperature, hybrid furnace at a small scale (to be fabricated through this research work) is not available in literature but some information regarding giant sized electric arc furnaces are available and used as a guide line in the design of this type of furnace. This hybrid furnace can be used in both gas and electrical modes whatever is appropriate and economical with easy handling. It will help in extracting metallurgical grade silicon with a purity range of 95-97 % (which can be further enhanced through other processes in order to make it compatible and suitable for international market). The present international price for metallurgical grade silicon is around \$2500-3000 / ton FOB depending upon the purity level [7], which could be reduced if produced locally while using existing facilities.

The outcome of this research work is anticipated to result in utilization of more than 200 million metric tons of quartz reserves of Pakistan and ensure value

added utilization of the raw material instead of other nominal uses while the large scale production may itself assist in controlling the cost of indigenous metallurgical grade silicon and solar cells production.

2. High temperature furnaces characteristics

Following characteristics are necessary to be incorporated for this furnace.

2.1 Adequate Strength

This furnace must have adequate compressive strength both at normal and high temperature to sustain the amount of heat produce. Moreover, it must have a sufficient dimensional accuracy and stability. Following properties has been checked /incorporated with regard to the strength;

- Refractoriness under load
- Compressive Strength
- Dimensional accuracy
- Dimensional stability (after expansion / contraction)
- Resistance to abrasion

2.2 Resistance to Destruction

There are two main forces which may act on refractories namely the chemical action of slags / dust / gases and the physical action of expansion / contraction that can lead to spalling. Following factors incorporated during the design of this furnace;

- Temperature developed inside the furnace
- Furnace atmosphere / environment
- Chemical composition of the refractory
- Porosity structure of the refractory
- Temperature gradient in bricks(govern depth to which molten flux can penetrate)
- Chemical composition and melting temperature of the fluxing agent
- Fluidity of the fluxing agent
- Rate at which fluxing agent is supplied
- Surface tension between flux and refractory (tendency of the flux to wet the refractory)
- Fluidity of the reaction product
- Rate at which the reaction product flows away from the face of the refractory (or removed in other ways)
- Agitation / turbulence of molten fluxing agent flowing down the face of the furnace wall (the turbulence is usually formed by gases moving at high velocity).
- Turbulence of slag floating on molten bath
- Convection currents

3. Design Calculation and Fabrication

To develop a small scale, high temperature, hybrid furnace for the extraction of Metallurgical Grade Silicon from raw mineral quartz, following

design parameters need to be evaluated in order to confirm the proper working and melting of required charge in the furnace.

3.1 Outer Shell Fabrication

Since the heating process can continue for more than two hours (to achieve a high temperature of 2000°C), a high grade mild steel plain carbon cold rolled sheet of 3.2 mm thickness (generally known as 8 gauge) conforming to AISI-1020 (American Iron and Steel Institute) is selected [8,9]. Table 1 shows chemical composition of 3.2 mm steel sheet while figure 1 shows different steps for developing of furnace outer shell. The bottom of the furnace shell is welded with 4 pedestal legs of 2x1/4 inches of angle iron with cross beams (for easy handling and erecting) as shown in figure 1-d [10].

Table 1: Composition of 3.2 mm Mild Steel Sheet

C	Si	Mn	S	P	Fe
0.17 - 0.23	0.15 - 0.35	0.6 - 0.8	0.03 Max	0.03	Rest

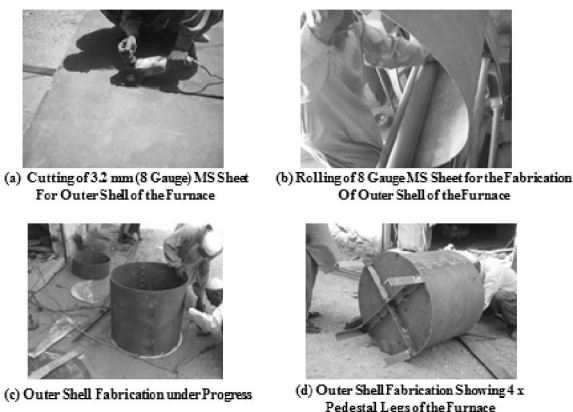


Figure 1: Furnace Outer Shell Fabrication Steps

In order to reinforce the outer furnace shell (to sustain high temperature developed inside the chamber), 4 mm thick (8 in numbers) Mild Steel flat strips welded outside of the shell as shown in figure 2-a & 2-b [11, 12]. These strips will also act as support to hold ceramic wool (external wool insulation) used to avoid heat dissipation in the atmosphere [13].

The top of the shell and lower edge of the cover are flanged by 10 mm thick MS flanges having external diameter of 765 mm and internal diameter of 705 mm. Four deep slotted holes of 12 mm diameter with 20 mm length have been drilled in the top cover of the flange to fasten the top cover with the shell

(figure 2-c). Four bolts of 12 mm X 100 mm thickness are permanently fixed by welding with the shell wall using pivot/swing mechanism to fasten shell and top cover (figure 2-d), while 180 mm diameter hole is made in upper shell of the top cover for the insertion of graphite electrodes (when fired electrically).

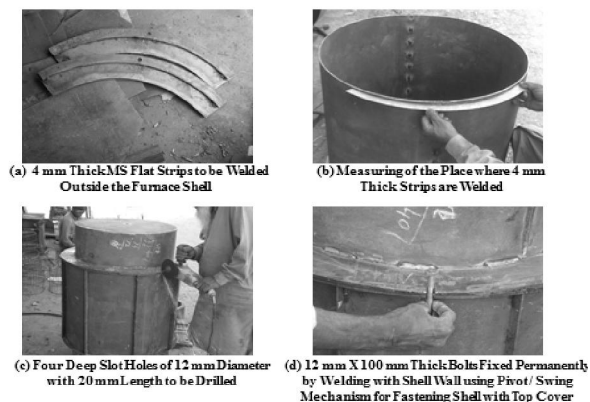


Figure 2: Furnace Outer Shell Fabrication Steps

3.2 Gas Firing window and Exhaust Chimney Fabrication

In order to provide passage for gas firing and exhaust chimney, two coaxial windows of 120 mm x 120 mm dimensions are opened in the furnace outer shell. The lower window is used as the opening port of the high velocity burner (figure 3-a) which is further extended and welded in angular state to fix the burner in such a way that its nozzle is direct tangential to the periphery of internal chamber of the furnace. The upper window as shown in figure 3-b is designed to act as a vent for the exhaust of hot flues gases through the chimney. An angle iron frame of 230 mm x 230 mm x 310 mm long has been welded surrounding coaxially at the upper window which will be used as flue channel with the chimney resting on the frame with fasteners. The channel frame is lined using hot face insulation and high alumina castable refractory while the outer wall of the channel is covered through welding of the MS sheet around it [14]. The chimney is fabricated by using 10 gauge MS sheet which is internally lined with hot face insulation bricks shown in figure 3-c.

The outer shell of chimney is also fabricated with the same 3.2 mm (8 gauge) MS sheet to sustain the high temperature exhaust gases. Only the interior (thicker part of the chimney) is lined with hot face insulation bricks to avoid the impact of hot gases on the outer shell of the furnace. The upper part of the chimney is again fabricated with 3.2 mm mild steel channel with canopy at the top to escape the exhaust gases in the atmosphere as shown in figure- 3-d.

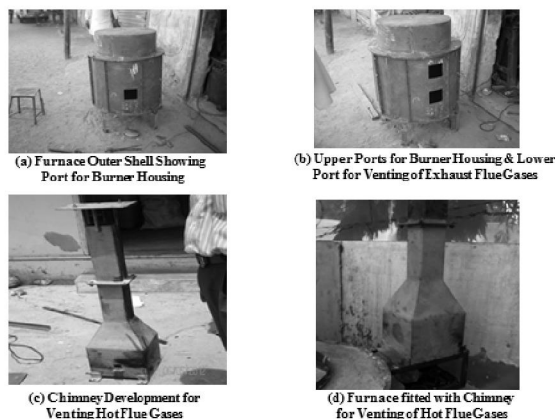


Figure 3: Gas Firing Window and Exhaust Chimney Fabrication

3.3 Inner Chamber Design

The inner chamber of the furnace need to be strong enough to sustain the high temperature developed inside and must ensure quick and safe melting. Generally one ton of molten steel occupies 0.145 m^3 , while the mass ratio of slag for acid lined furnaces may be taken equal to $0.03 - 0.04$ [15].

The internal diameter to depth ratio of the chamber is taken as:

$$H_1 = 1.5 D \quad (1)$$

Where; D = Inner chamber diameter,

H_1 = Height (depth) of the chamber.

Normally the existing furnaces are having the height (H_2) roughly equal to 4 times of the total chamber depth;

$$H_2 = 4 H_1 \quad (2)$$

Thus the total volume of the chamber (V_c) can be calculated as;

$$V_c = H_1 \times D^2 \text{ (in } \text{m}^3\text{)} \quad (3)$$

The banks of a furnace are usually made in such a way as to avoid the inner chamber heat dissipation and to ensure that the slag does not contact the brick work or reach the joint between the wall blocks and banks [15].

The total height of the roof above the inner chamber level (H_t) can be calculated as;

$$H_t = H_1 + 2.5 H_1 \quad (4)$$

The inner chamber design of the furnace is based on to cover the following;

- The gas firing process
- The electric arc process

The inner chamber dimension keeping above in view, are kept $240 \text{ mm (D)} \times 360 \text{ mm (H}_1\text{)}$. The inner chamber cavity is to accommodate two to three graphite crucibles containing test sample with reactants materials, placed one over the other separated through the lids of the crucible (The lids

would develop internal pressure of gaseous products necessary for solid phase reaction). This solid phase reaction would be carried out in enclosed crucible heated through high velocity natural gas which will force high pressure air combination burner to produce extreme combustion conditions (specially designed to feed pure oxygen) to assist combustion to attain high temperature beyond 1600°C . (The burner would be firing tangential to the periphery of the internal firing chamber).

The liquid phase reaction would be carried out using electric arc through the submerged graphite electrodes producing an arc of $> 3000^\circ\text{C}$ which will trigger the localized reaction. This high temperature arc will also help in bringing the reaction product (Metallurgical Grade Silicon) into liquid state to settle at the bottom of the reaction vessel (graphite crucible).

3.4 Inner Chamber Fabrication

In order to develop a high temperature hybrid furnace which can sustain high temperature (produced inside without affecting the chamber walls), suitable refractory material along with bricks lining is required. Table 2 shows different characteristics of various types of brick lining and refractory materials. To develop inner chamber, the base of the furnace is paved with high alumina fire clay mortar added with grog to sustain the high temperature developed [16, 17, 18] as cleared from figure 4-a. A layer of $4/5$ inch of high alumina insulation bricks are then placed over this fire clay mortar as shown in figure 4-b. The bricks are then further paved with the high alumina mortar in order to make the inner wall strengthened and solid [19] as shown in figure 4-c. The next inner chamber of the furnace comprised of two main layers of calcined bauxite and fuzed alumina (corundum) which is directly facing the internal chamber of the furnace [20] cleared from figure 4-d.

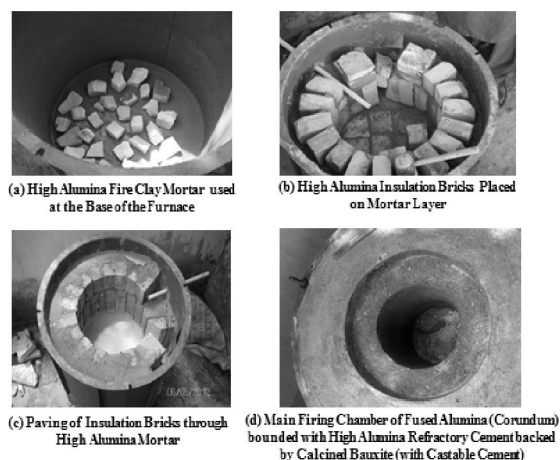


Figure 4: Furnace Inner Chamber Development Stages

Table 2: Characteristics of Different Refractory Materials for High Temperature Furnaces [16, 17, 18, 19]

Name	Type	Mullite	Bonded Fused Alumina	Fused Alumina	Fused Alumina
Analysis	SiO ₂	29.39	0.6		
	Al ₂ O ₃	68.26	99.2	90-99.1	88-90
	TiO ₂	0.85			
	Fe ₂ O ₃	0.62			
	Mg	0.01			
Others	CaO	0.16			
	Na ₂ O	0.84			
Refractoriness	Cone	38	42	38-41	>42
	^o C	1850	2000	1850-1900	2050
Specific Heat				0.174-0.304	0.272 (20-800 ^o C)
Apparent Porosity		20-30	20-30	19-23	21
True Porosity					
Water Absorption		2.36-2.38	3.06	3.0	3.0
Bulk Density (g/cm ³)		148	191	187	187
	True Specific Gravity			3.8 / 4.0	3.90
Cold Crushing Strength (Kg / cm ²)		598			
Modulus of rupture (Kg / cm ²) x 10 ⁻⁶		ASTM 133-49 127			
Mean Reversible Thermal Expansion		5.76		7.0 (20-1000 ^o C)	8.1 (21-1000 ^o C)
Thermal Conductivity	c.g.s		>0.0006	18-20 (1100 ^o C)	
Linear Change on re-firing	Temp-Time	1700		1500	1500
	5 Hrs			Nil	Nil
Refractoriness Kg / cm ²	Under Load		1500 / 1700	1500	1500
	Temp	1590	0.0 / 0.1	-	-
	% Deformation	1.8		1-2	2
Thermal Shock Resistance			Medium		
Spalling Loss %					Spalling in dia 60-70
Permeability					

Complete solid shape of inner firing chamber of the furnace is shown in figure 5-a & b. The top cover of the furnace is also built by using the same material (used in the furnace main chamber development). This will enable in sealing of the internal chamber of the furnace thus allowing maximum sustainability of high temperature produced. Figure-5 c & d show development process of furnace cover.

4. Design and Development of Temperature and Pressure Measurement System

4.1 Thermocouple Development

A special type thermocouple is required for the measurement of high temperature (up to 2000^oC), which was not readily available in the market. This difficulty was overcome through acquiring different thermocouple measuring parts like thermocouple wires (one pure platinum wire while other 13 % Rhodium / Platinum wire), ceramic beads, ceramic tubes and head / junction boxes as shown in figure 6-a

& b. These items were assembled to make the unit of S-type of thermocouples, which can measure the smallest temperature fluctuation (when it is connecting with the digital multimeter as shown in figure 6-d) with excellent thermal efficiency / performance. The performance of different type of thermocouples is shown in figure 7 [21]. These thermocouples are inserted / placed in the chamber through a hole provided during the furnace fabrication process for easy access to the inner chamber during firing as shown earlier in figure 4-b. A table is consulted for the conversion of digital multimeter readings in to the temperature attain during the firing [22].

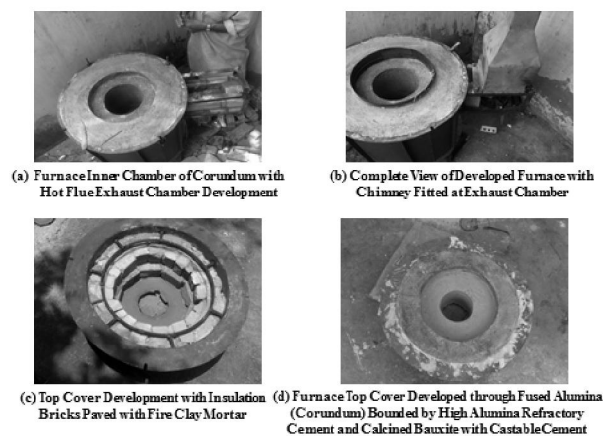


Figure 5: Complete Furnace Inner Chamber and Top Outer Cover Development

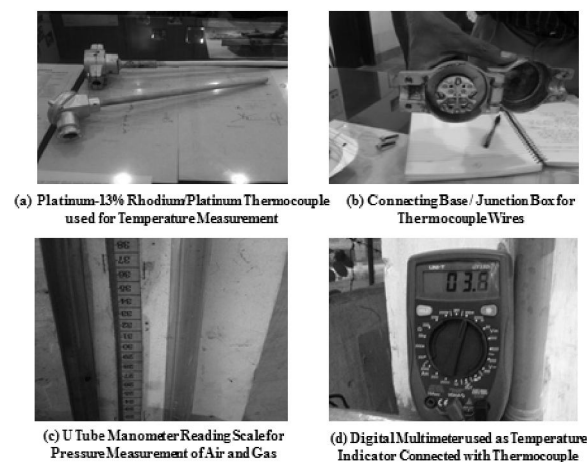


Figure 6: Development of Thermocouple and Temperature Measurement Scale

4.2 Air and Gas Pressure Measuring System

In order to measure the air and gas pressures, two separate sets of U-tube manometers are fabricated through acquiring different components like plastic tubes, aluminum angles, wooden strips as base for

holding these items and measuring scale (in mm calibration). These parts were assembled together in order to provide proper and fast measurement of pressure are shown in figure 6-c. The water column reading can be seen on these manometers tube, which shows air and gas pressures.

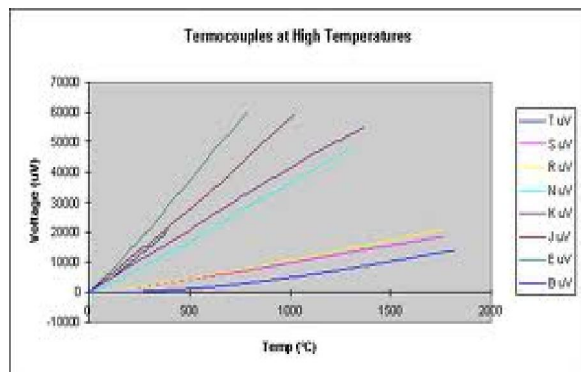


Figure 7: Thermocouples performance at different temperatures

5. High velocity induce burner development

In order to fire the furnace with natural gas or liquid petroleum gas at such a high temperature (around 1800°C), a specially design high velocity induced burner is developed keeping following in view [23, 24, 25, 26];

- Operational parameters
- Fabrication parameters

5.1 Operational Parameters

The operational parameters of this high velocity gas fired induced burner with both natural gas and liquid petroleum gas are shown in Table 3;

Table 3: Operational Parameters of High Velocity Gas Fired Induced Burner

Fuel Type	Density	Calorific Value (Gross)	Calorific Value (Net)
Natural Gas	0.71 Kg / m ³	8800 Kcal / Nm ³	8500 Kcal / Nm ³
LPG	2.2 Kg / m ³	26200 Kcal / Nm ³	11920 Kcal / Nm ³

5.2 Blower Selection for Combustion air

Combustion air for high velocity burner to be fed through a powerful blower (shown in figure 8-a & b) with the following configuration [27];

- Blower Type Multi Stage series
- Power 2.3 KW

- Static Pressure 1000 - 1200 mm of Water Column
- Flow Capacity 300-400 m³/ hr
- Flow Medium Air
- Temperature 16°C - 30°C
- Density 1.22 Kg / m³

The oxygen gas will be fed at peak stage of firing and it will assist combustion process in order to attain more than 1600°C of temperature (The density of oxygen at Standard Temperature and Pressure is 1.42 Kg /m³).

The high velocity burner may be firing at 50,000-80,000 Kcal/hr during peak stage of firing which may also be determined during a test firing. However the burner has the capacity to consume 10-12 m³/hr of natural gas and 150-200 m³/hr of combustion air during peak stage of firing. The combustion may be further assisted with pure Oxygen (fed at peak stage), while liquid petroleum gas (LPG) can be used as an alternative / additional fuel in case of insufficient natural gas.

5.3 Fabrication of High Velocity Burner

High velocity burner includes gas nozzle with 18 holes comprising 3 rows (with 2 mm diameter each) as cleared from figure 8-c. These holes are provided round the periphery of the nozzle in order to direct gas normal to the periphery of the nozzle (figure 8-d).

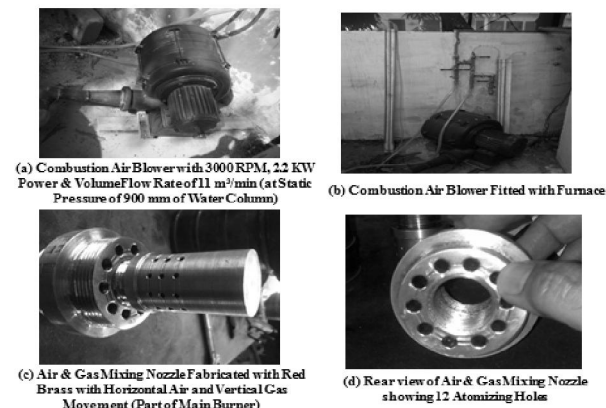


Figure 8: Fitting of Combustion Air Blower and Burner Nozzle Development

5.4 Fabrication of Air and Gas Mixing System

Complete air and gas maxing system including nozzle cap and air and gas mixing housing is shown in figure 9 (a-d). This is fabricated through red brass (to confine air and gas mixture at a very high pressure and temperature) which will provide its passage up to the tip of the burner directly into firing chamber.

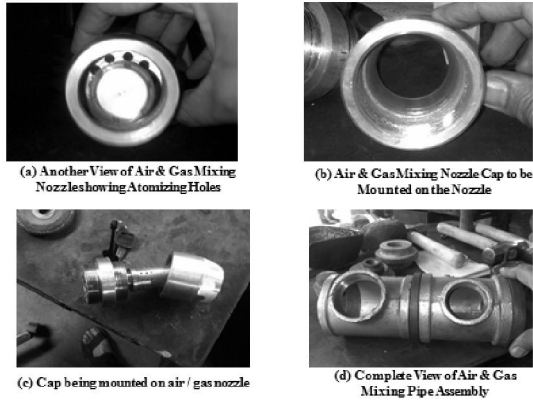


Figure 9: Burner Nozzle / Cap and Brass Housing Fabrication

6. Fabrication of electrical firing system

In order to fire the furnace electrically, following parameters need to be incorporated;

6.1 Selection of Transformer

Selection of the transformer plays an important role as far as electrical firing of the furnace at such a high temperature is concern. In this regard following parameters were incorporated;

- Voltage required by the load
- Amperes or KVA required
- Frequency in Hz
- Single phase / Three phase
- Supply voltage at the source

For this type of furnace, a transformer is selected as per following specifications;

- Primary side voltage 440 V
- Primary side Ampere 30 A
- Secondary side Voltage 40-50 V
- Secondary side Ampere 250-350 A

Following formula will be used for determining three phase KVA transformer (when Volts and Ampere are known);

$$\begin{aligned}
 \text{KVA} &= \text{Volts} \times \text{Amperes} \times 1.75 / 1000 \\
 (1) & \\
 &= 240 \times 30 \times 1.75 / 1000 \\
 &= 125 \text{ KVA}
 \end{aligned}$$

Hence, a transformer with 125 KVA power is suitable to be used with this type of furnace as shown in figure 10-a [28]. Table 4 shows few recommendations as far as transformer power for different size of the furnace capacities are concern.

The unavoidable switching off of the furnace during the melting process necessitate pushing of the scrap, operating furnace at a restricted / reduced voltage when arcs are open and radiation of high heat onto the walls and roof, the average power consumed

during the melting period can be found by using a factor of 0.8 approximately as [29, 30];

$$P_{av} = 0.8 P_{ap}$$

Now the useful power consumed during the melting period can be represented as;

$$P_u = P_{av} \cos \phi \eta \text{ el (KW)}$$

The upper voltage (secondary voltage) attain for small furnaces can be selected by using the following empirical formulae;

$$V = 15 (P_{ap})^{1/3} \quad (\text{For Basic Furnaces})$$

$$V = 70 + 15 (P_{ap})^{1/3} \quad (\text{For Acid Furnaces})$$

Table 4 shows recommended transformer power for different sizes of furnace [31];

Table 4: Recommended Transformer Power for Different Sizes of Furnaces

Transformer Power MVA			
Furnace Capacity (M.T)	Alloy Steel	Carbon Steel	Present Furnace
25	5-18	18-22	-
50	50-25	28-32	40
75	-	30-45	50
100	30-35	40-50	60
150	-	45-60	-
200	55-70	60-80	-
250	-	90	-
400	-	120	-

6.2 Electrodes selection

In order to achieve high temperature 2x graphite (carbon) electrodes are used, which are inserted inside the chamber through the clip holders as clear from figure 10-b. These electrodes need to be used with extra care as they are expensive and so its usage can increase over-all cost of MGS production.

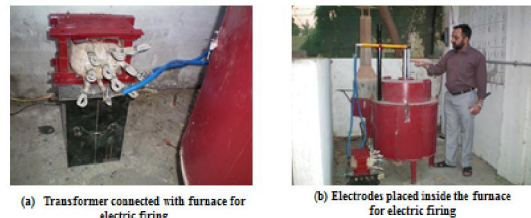


Figure 10: Furnace electrical firing

7. Conclusion

Pakistan is blessed with large deposits / reserves

of quartz, which can be used for the extraction of metallurgical grade silicon. Unfortunately in Pakistan this precious mineral is not being used up to its actual potential and either is being used for nominal tasks or being imported to other countries. Due to recent development as far as electronic and solar cell manufacturing through the utilization of silicon is concern; there is a dire need to establish cheap methods of producing / extracting silicon from this mineral. This will avoid dependence on outside world as exporting of silicon from other countries will be minimized and solar cell manufacturing can be established in the country. Moreover, the price of silicon in the region will be cut down to acceptable level as current price is 2500-3500 US \$ / ton.

Due to the electricity crises, the shift of electricity production from conventional methods to alternate / renewable energy production methods has been seen world widely. In fact solar option is one of the best options as far as renewable energy production is concern and in order to go for this option it is necessary to establish solar cell manufacturing industries thus indigenizing solar cell manufacturing. This can only be done if Pakistani industry will be able to produce silicon locally through acquire safe and cheap methods / procedures.

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