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The Manufacturing of Metallurgical Grade Silicon from Diatomaceous Silica by an Induction Furnace

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***Abstract*—**The metallurgical grade silicon (MG-Si) is obtained from the reduction of silica (SiO_2) in an induction furnace or an electric arc furnace. Impurities inherent in reduction process also depend on the quality of the raw material used. Among the applications of the silicon, it is used as a substrate for the photovoltaic conversion of solar energy and this conversion is wider as the purity of the substrate is important. Research is being done where the purpose is looking for new methods of manufacturing and purification of silicon, as well as new materials that can be used as substrates for the photovoltaic conversion of light energy. In this research, the technique of production of silicon in an induction furnace, using a high vacuum for fusion. Diatomaceous Silica (SiO2) used is 99 mass% initial purities, the carbon used is 6N of purity and the particle size of 63µm as starting materials. The final achieved purity of the material was above 50% by mass. These results demonstrate that this method is a technically reliable, and allows obtaining a better return on the amount 50% of silicon.

***Keywords*—**Induction, amorphous silica, carbon microstructure, silicon.

I. Introduction

THE sun is the most available energy source considered renewable and clean. Silicon is the element most used for the conversion of solar energy into electrical energy.

Solar cells are the technology corresponding to the problem solution of energy in our planet. This technology is already quite usual for aerospace applications [1]. However, for their application in terrestrial areas, they are still limited due to the high cost of photovoltaic's modules [1], [2]. Research is underway in some countries, with new research methods aimed for the manufacture and purification of silicon or new materials which can be used as a substrate for energy conversion, always looking for a reduction in the cost of production photovoltaic devices [1]-[3]. With the increase in the demand for power generation of solar cells, a large amount of solar grade silicon should be placed on the energy market. Diatomaceous earth is considered one of the promising resources of silicon. It essentially contains amorphous silica in diatoms frustules, silica can be readily purified by dissolving in an alkaline solution. In addition, there are several reserves are large enough around the world, but the price of a tone of diatomite costs a little more than a tone of sand [4]. This paper deals with the results obtained following carbothermal reduction of diatomaceous silica.

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II. Experimental Method

A. Materials

Amorphous Diatom silica is prepared from Algerian diatomite earth according to [5], (Fig. 1) shows the diatomite deposit Site and Fig. 2 show SEM image of diatomite, purity of silica is about 99,43% SiO_2, it chemical composition is given in Table I. The carbon used in this experiment have a particle size of 63 µm, the diatomaceous silica is mixed with carbon, polymer and water to a granulating the mixture.

The crucible used in these experiments with a size of 20cm in height and 10 cm diameter, and the furnace used in the experiments is shown in Fig. 3 (a).

TABLE I
Chemical Analysis for Raw Diatomite and Purified Silica Diatomite

Elements	Raw diatomite at%	Purified silica at%
Ti	ND	0,07
Mg	0,05	0,11
K	0,14	ND
Fe	0,14	ND
Mn	ND	ND
Cl	0,26	0,11
Al	0,40	0,14
Ca	0,34	ND
Ba	0,17	ND
S	1,62	0,07
Cu	0,83	ND
Na	9,98	0,07
Si	86,07	99,43
total	100	100

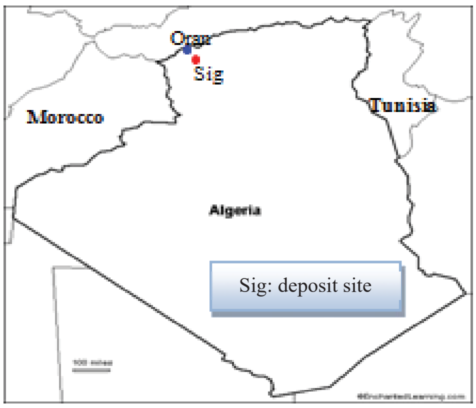

Fig. 1 The location of the diatomite deposits

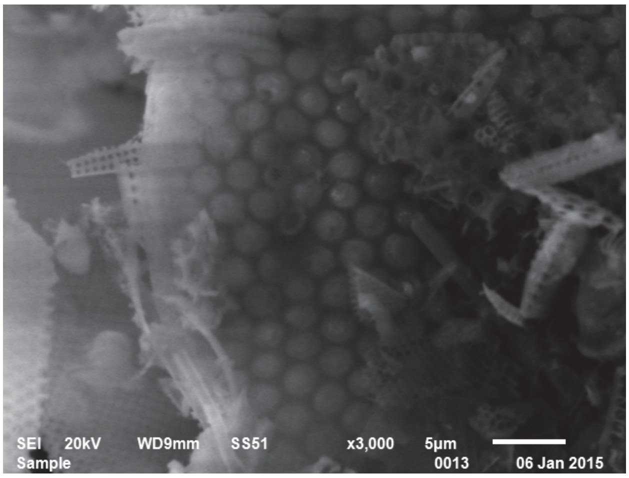

Fig. 2 SEM image of diatomite

(a)

(b)

Fig. 3 (a) Image of induction furnace, (b) Image shows the inside of the furnace

The Principe of induction heating is mainly based on two well-known physical phenomena, Electromagnetic induction and Joule Effect. For electromagnetic induction, it can say that the principle is very easy, the crucible is put inside a coil, an electric current flows through the coil. So, this current will create a magnetic field, this last create a current named Eddy current on the surface of the crucible Fig. 3 (b). Shows the inside of the furnace component

It is known that when the current flows through a conductor, the electrical energy will be converted to a thermal energy by Joule effect. Table II lists the experimental conditions.

III. Characterization

A. Scanning Electron Microscopy (SEM)

The structures and the surface morphologies of the obtained powder samples were examined by scanning electron microscope (TM3000, HITACHI, NIMS, Tsukuba, Japan). The samples were observed and analyzed at an 8.3mm working distance and 15KV acceleration voltage.

B. X-Ray Diffraction

Low angle X-ray diffraction patterns were obtained in reflexion mode (θ -2 θ) using PANalytical EMPYREAN diffractometer (USTO-MB, Algeria), employing CuKα radiation (λ=1.5406 Å), data was collected between 2θ=10° and 90° and 100°.

C. Inductively Coupled Plasma ICP-AES

Samples of silicon undergo a decomposition process before analysis. *Instrumentation, operating conditions and measurement* (ICP-AES): Horiba ULTIMA 2 were used. Institutions de measure: Foundation, Iwate Prefecture sud Technology Research Center

IV. Results and Discussion

A. Silicon Elaboration

Diatomaceous silica was mixed with micro-structured carbon using a polymer to create particles sizes of the order of centimeters, the goal is that the mixture does not escape during the vacuum process. Silicon was prepared under condition mentioned, Table II lists the experimental conditions, the carbothermal reduction of silica inside the furnace, occurs in three different stages as described by the equations below:

$$SiO_2 + C = \underline{SiO} + CO \quad (1)$$

$$\underline{SiO} + 2C = SiC + CO \quad (2)$$

$$SiO + SiC = 2Si + CO \quad (3)$$

TABLE II
Experimental Conditions for Experiments

Ar pressure	0.07 MPa
Voltage	230V
T° Maximum	~1650 ℃
Molar ratio SiO_2:C	3:2
Time	15 min

The deposition of the charge in the crucible in the form of successive layers is described in Fig. 4.

B. Chemical Analysis for Purified Diatom (EDX)

The chemical composition of silicon from diatom silica was characterized using EDX, and the complete composition analysis can be found in (Table III), it means that the material elaborated contain silicon but also the silicon carbide.

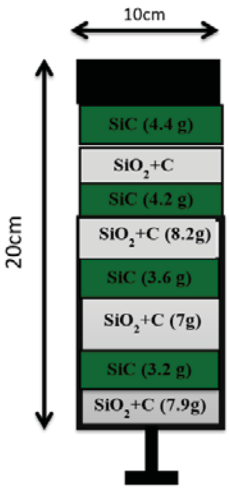

Fig. 4 Image described the arrangement of the load in the crucible SX1744

TABLE III
CHEMICAL COMPOSITION OF METALLURGICAL SILICON WAFER OBTAINED

Element	Weight %	Atomic %
Carbon	40.285	59.859
Oxygen	5.033	5.614
Sodium	0.023	0.018
Magnesium	ND	ND
Aluminum	1.535	1.015
Silicon	52.478	33.347
Sulfur	ND	ND
Potassium	ND	ND
Titanium	ND	ND
Manganese	0.119	0.039
Iron	0.139	0.044
Zirconium	0.191	0.037
Barium	0.196	0.025

C. X-Ray Diffraction

The XRD patterns of metallurgical silicon is given in (Fig. 6), it shows an essentially crystalline phase in all the samples, they contain Silicone (Si) and Silicone Carbide (SiC). The sample named SX1744(8 layers) is silicon from diatomaceous silica it is compared to other samples containing nickel SX1743 and the sample named SX1742 contain only (2 layers). Every peak is related to one special component (distinguished by the diffraction angle 2 theta) as shown below.

Fig. 5 Image of metallurgical silicon wafer obtained SX1744

TABLE IV
CONCENTRATIONS OF CHEMICAL ELEMENTS IN OBTAINED SILICON

Sample name	SX1744 ppm
Al	0.114
B	6
Ca	453
Cu	<0.2
Fe	317
Mg	50
Ni	0.0293
P	<0.1
Ti	41
V	<0.3
Zn	<0.2

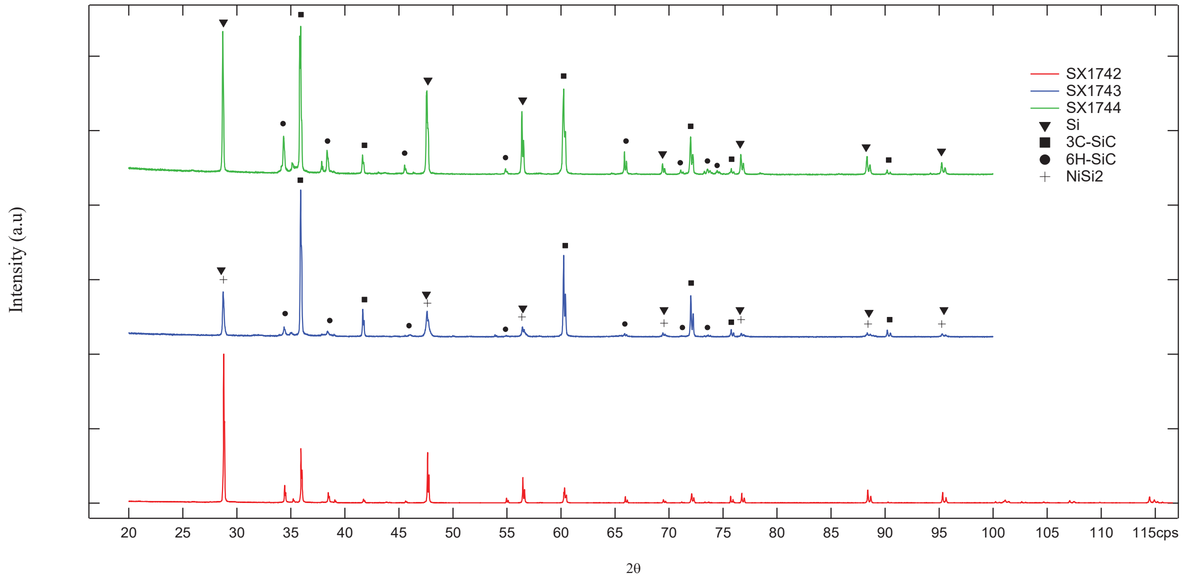

Fig. 6 XRD patterns of silicon obtained from the carbothermal reduction of silica of diatomaceous earth SX1744

D. Inductively Coupled Plasma ICP-AES

Measurements of each element were quantified by absolute calibration curve method. It shows the results of the analysis, also shown in detail in index Table IV. The residue rate: 52%

V. Conclusion

The objective of this work is to produce metallurgical silicon from Algerian amorphous diatomaceous earth as an abundant source of silica, to integrate the application of silicon in Algerian photovoltaic industry as raw material, knowing its abundance in the Earth's crust. The metallurgical silicon can be successfully prepared from Algerian diatomite silica through carbothermal process using induction furnaces. Until now the result achieved, after following consecutive steps on the furnace is 48%.

- Molar Ratio: the best Molar ratio for getting silicon (Si) is the mix (SiO_2: C=3:2).
- Max Temperature: The best temperature for getting silicone is between 1350-1650°C.
- The catalyst Adding: Adding a catalyst to the reaction minimize the energy consumed by the reaction, this, by comparing of the sample SX1744 with the references samples.

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