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Synthesis of $MgFe_2O_4$ spinel using steel waste as iron resource

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Abstract*— This project is immersed in the existing world problems on the need of substituting the coatings in the clinker producing ovens in the cement companies that are currently of chrome-magnesite due to the fact that they are potentially pollutant since they release during their hexavalent chrome to the environment. A viable option is the incorporation of spinel blocks since they comply with the necessary requirements of a refractory material and they do not pollute. These spinel blocks are made using as a raw material the iron residue of the "Antillana de Acero" iron plant, where 60 tons a day are produced. We incorporate to this residue the mineral called cyanite without magnetic separation from the Las Nuevas, Isla de la Juventud where there is a 100,000 ton reserve. Both raw materials are located in Cuba.

***Index Terms*—Ferrite spinel, Steel waste, Cianite, Ceramic.**

I. INTRODUCTION

Glass ceramics are polycrystalline solids that usually contain a residual vitreous phase. They are obtained from the fusion and solidification of soluble inorganic vitreous components that are susceptible to controlled crystallization [1]. The glass ceramic processes is being applied in the recycling of mineral residues of mines and industries such as goethite sludge's of the hydrometallurgical industries [2] and of geothermal plants and have been used to produce ceramic materials [3], glass ceramics [4], and glasses [5], compound materials [6], ashes and optic type glasses [7]. The name spinel is parallel used as a name of chemical compound – $MgAl_2O_4$ and as a name of the group of chemical compounds with general formula – AB_2O_4, where A and B describe respectively, bi- and tri-valent metals. Stoichiometric spinel contains 28.2 wt. % of MgO and 71.8 wt.% of alumina. Its melting temperature is 2135°C [8].

The spinel group encompasses a large number of aluminates, gallates, ferrites, titanates and solid solutions of these groups. None of these minerals, when melted and cooled, forms a glass. It is possible, however, to precipitate many of these phases from a glass consisting largely of silica and a considerable percentage of spinel chemical components [9]. Ferrites have been synthesized by various methods such as solid-state reaction, co-precipitation, high-temperature self propagating, micro emulsion, solvo thermal, mechanosynthesis, hydrothermal, sol–gel and combustion techniques [10-14]. Spinel ferrites are of scientific and technological importance in recent years due to both their unique properties and broad range of applications in diverse areas such as magnetic recording and separation, ferro-fluid, magnetic resonance imaging (MRI), biomedicine, catalyst, etc. [15–17].

II. EXPERIMENTAL PROCEDURE

Two compositions (Table 1) were chosen according to ternary diagram system: FeO-Al_2O_3-SiO_2 to synthesis ferrite spinel using as raw materials mineral cyanita as source of Al_2O_3 and SiO_2 obtained from the mine Las Nuevas in the Isla de la Juventud, Cuba. To provide the iron in the synthesis of ferrite spinel was used an iron waste that is obtained as a sub product of steel aspersion obtained from the steel industry of Cuba. Both raw materials were characterized in terms of chemical analysis.

Material	SiO_2	Al_2O_3	FeO
GC – 1	42	16	42
GC - 2	26	09	65

Table 1. Chemical composition (w/w %) according to ternary diagram SiO_2-Al_2O_3-FeO

The chemical composition of the starting raw materials, i.e. cyanite and iron residue are given in Table 2. The cyanite used in this investigation contains 47 wt.% SiO_2 and 46.86 wt.% Al_2O_3. Minor amount of impurities like MgO, CaO, TiO_2 is also present as shown in Table 2. Iron residue contains 5.68 wt.% SiO_2, 1.16 wt.% Al_2O_3, 40.42 wt.% Fe2O3 and 36.48 wt.% FeO, Other compounds are present in minor amount like Cr_2O_3 at 1.53 wt.%. NiO at 0.05 wt%, CuO at 0.22 wt.% and ZnO at 2.08 wt.% in the iron residue and not presented in the cyanite. The raw materials were carefully mixed in the crucible with 30 mL of deionized water the precursor glasses mixtures were placed in the furnace at a temperature of 1300 °C for 120 min, after this time the melted glasses were cast in cold deionized water to solidify. Precursor's glasses were subsequently placed at heat treatment at; 1) 650 °C/240 min. and 2) 1088 °C/600 min for GC-1 and 1170 °C/600 min for GC-2 with heating and cooling rate of 3 °C/min. The glasses were characterized by Scanning Electron Microscopy (SEM) using a JEOL JSM 7600F microscope and phase analysis was carried out by X-ray diffraction, The X-ray diffraction (XRD) patterns were collected on a Bruker modelo D-8 advance Diffractometer.

Table 2 Chemical composition (w/w %) of raw materials

Oxides	Iron Residue	Cyanite
SiO_2	5.68	47
Al_2O_3	1.60	46.86
Fe_2O3	40.42	4.39

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CaO	7.64	0.11
MgO	4.23	0.10
TiO_2	--	0.63
FeO	36.48	--
Cr_2O_3	1.50	--
NiO	0.05	--
CuO	0.20	--
ZnO	2.00	--
Ignition Lost	--	0.90

III. RESULTS AND DISCUSSION

A. X-Ray Diffraction

In the XRD spectra (Figure 1a) treaty at 650 °C/240 min the predominant crystalline phase is $MgFe_2O_4$ in GC-1 glass which has 79 wt.% of iron residue in its composition which is used as iron sources. Figure 1b show the XRD diffractogram of GC-2 glass which has 92 wt. % of iron residue in its composition, the first thermal treatment at 650 °C/240 min promote the formation of iron-magnesium spinel.

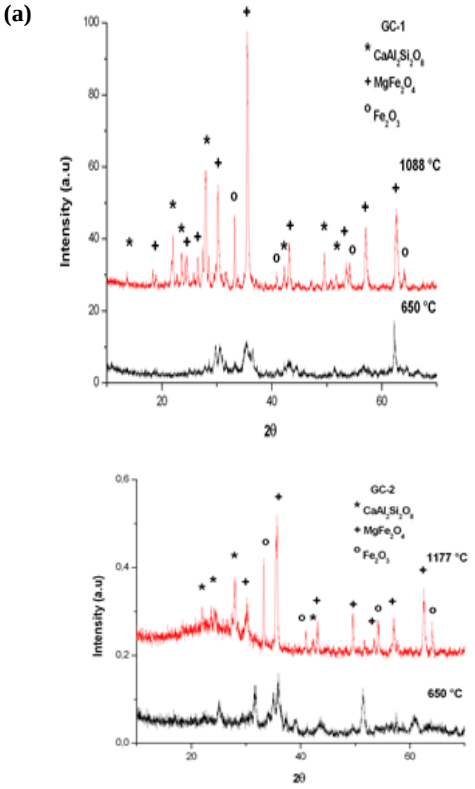

Fig 1. X-ray diffraction patterns of annealed (a) GC-1 treated at 650 °C/240 min. and 1088 °C/600 min. (b) GC-2 treated at 650 °C/240 min. and 1170 °C/600 min.

To decrease this vitreous dome a second thermal treatment was applied for sample GC-1 was applied at 1088 °C/600 min. an the XRD pattern of GC-1 show in Fig. 1a we can see the crystalline ferrite spinel ($MgFe_2O_4$) phase was defined more clearly and predominate against the ortho-silicate phase as Anorthite ($CaAl_2Si_2O_8$). In the case of GC-2 was treated at 1170 °C/600 min after that was tacked is XRD pattern show in Fig. 1b. clearly we can see that the vitreous dome persist between 15° and 35°, moreover the crystalline phase presented in GC-2 at 650 °C change with the second thermal treatment. The new crystalline phase found in the GC-2 sample was ferrite spinel phase ($MgFe_2O_4$) and the Anorthite phase ($CaAl_2Si_2O_8$) which are present in sample GC-1 from the first thermal treatment and clearly define these crystalline phases with thermal treatment at 1088 °C.

B. SEM characterization

The surface morphology of GC-1 and GC-2 glasses were characterized by scanning electron microscopy (SEM). Figure 2 show the corresponding micrographic of GC-1 material at 1088 °C which shows a homogeneous crystallization of ferrite spinel ($MgFe_2O_4$) on a crystalline phase composed of anortite ($CaAl_2Si_2O_8$), through analysis by EDS, were clearly identified chemical compositions of the phases present.

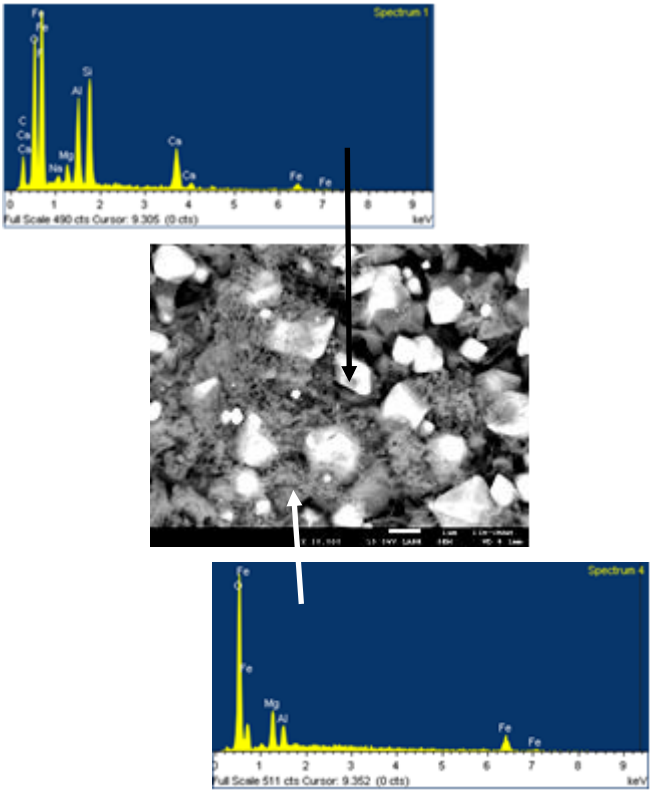

Fig 2. SEM/EDS Micrograph-Spectra of GC-1 annealed glass at 1088 °C/600 min.

The glass-ceramic material containing more iron waste (GC-2), presents a more abundantly the presence of ferrite spinel ($MgFe_2O_4$) phase and was diminished Anorthite ($CaAl_2Si_2O_8$) phase. The EDS analysis, confirm the chemical elements in each area. The Figure 3, present a micrograph and EDS spectra's.

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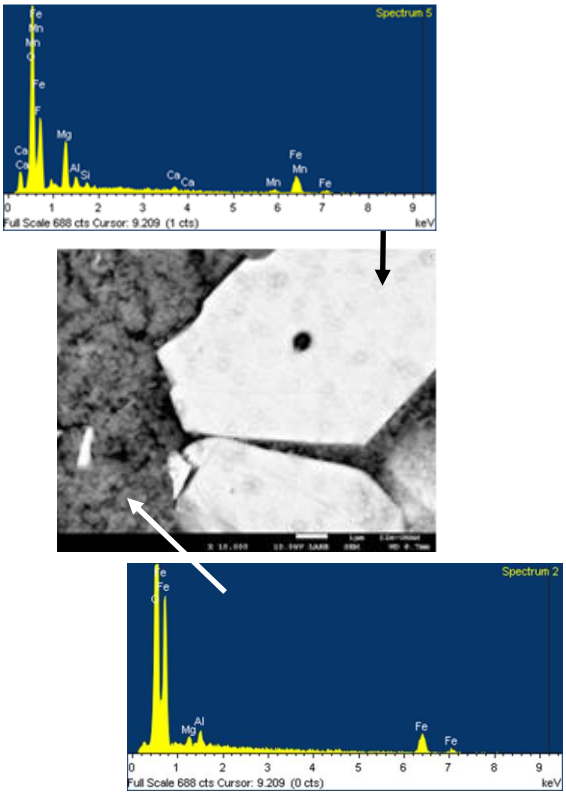

Fig 3 SEM/EDS Micrograph-Spectra of GC-2 annealed glass at 1170 °C/600 min.

IV. CONCLUSIONS

The use of this waste as raw material can be estimated within the material elaboration process where the base crystalline phase is Magnesioferrite Spinel [$MgFe_2O_4$]. The industrial waste generated in the steel industry is composed mainly of Zn, Si, Ca, Cr, Mg, Fe, S and Cl ions, where Fe is the predominant one. Through X-Ray Diffraction it is possible to clearly identify the predominant crystalline phases: Magnesioferrite [$MgFe_2O_4$] and Anorthite [$CaAl_2Si_2O_8$], the first one of the estimated formula and the second one as a product of the reaction of the industrial grade raw materials with the industrial waste composition. The generation of an additional phase was based in the GC-1 and GC-2; Hematite [Fe_2O_3], which can be attributed to a the solubility of the existing crystalline phases by increasing the temperature and/or that the residual vitreous phase tends to crystallize since it is soluble in it. Thermal studies of these materials are achieved after applying thermal treatments that will promote fusion at 1450°C/60 min., nucleation at 650 °C/240 min. and crystallization at a subsequent treatment at 1088°C with a 600 min crystallization for GC-1 and 1170°C for GC-2, with the purpose of increasing the degree of deglassification. It was possible to identify the adequate time-temperature relationship in order to carry out the crystallization process, which is of vital importance in processes involving energy output; It has been possible to utilize the residues generated in the steel industry.

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REFERENCES

[1] Z. Strnad, "Glass Science and Technology 8". Elsevier, pp. 76, New York, 1996.

[2] D. Valenzuela, C. Diaz, M. Avalos-Borja, Engineering Materials, 132-136, 2284-2287.

[3] C. Diaz, F.J. Valle-Fuentes, M.E. Zayas, M. Avalos-Borja, Am. Ceram. Soc. Bull., 78, 62-64, 1999.

[4] C. Diaz, F.J. Valle-Fuentes, Ceram. Soc. Bull., 78 [8], 112-115, 1999.

[5] C. Diaz, H. Gracia, Ma. E. Zayas, F.J. Espinoza, F.J. Valle-Fuentes, Am Ceram. Soc. Bull., 78 [3], 57-59, 2000.

[6] M. Romero, J. Ma. Rincon, S. Musik, V. Kozhukharov, Material Research Bull., (1999), 34 [7], 1107-1115.

[7] Ma. E. Zayas, F.J. Espinoza-Beltrán, M. Romero, J. Ma. Rincón, J. Mat. Sci. Letters, 17, 1099-1102, 1998.

[8] Muan, Osborne: Schlackenatlas. Verlag Stahleisen. mbH, D¨usseldorf, 1981, p. 30.

[9] F.J. Torres, J. Alarcon , J. Non-Crys. Sol. 320 (2003) 246–254.

[10] D. Chen, D. Chen, X. Jiao, Y. Zhao, M. He, Powder Technol. 133 (2003) 247.

[11] V. Sepelak, M. Menzal, I. Bergman, M. Wiebcke, F. Krumeich, K.D. Becker, J. Magn. Magn. Mater. 272 (2004) 1618.

[12] D.H. Chen, X.R. He, Mater. Res. Bull. 36 (2001) 1369.

[13] J. Fang, N. Shama, L.D. Tung, E.Y. Shin, C.J.O. Connor, K.L. Stokes, G. Caruntu, J.B. Wiley, L. Spinu, J. Tang, J. Appl. Phys. 93 (2003) 7483.

[14] Y. Shi, J. Ding, X. Liu, J. Wang, J. Magn. Magn. Mater. 205 (1999) 249.

[15] Raj K, Moskowitz R, Casciari R. J Magn Magn Mater 1995; 149:174–8.

[16] Gupta AK, Gupta M. Biomaterials 2005; 26:3995-1721.

[17] Stevens PD, Fan JD, Gardimalla HMR, Yen M, Gao Y. Org Lett 2005; 7:2085–8.

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I. It has following his start in the area of Development of ceramic nanostructured materials with applications in the areas of engineering, environment and health, has created a new line of research on use of industrial waste inorganic composition in vitreous and ceramic systems.

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