MICROSTRUCTURAL AND THERMAL CHARACTERISTICS OF THE SINTERED Al-Fe_2O_3 COMPOSITES

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Abstract:

This work has as an objective a study of evolution of characteristic properties of crystalline microstructure and mechanical hardening of aluminum by iron oxide (III), (hematite α-Fe_2O_3) nan energetic material known as thermite, samples of massive alloys, Al (base)-X wt% Fe_2O_3 (X =2, 4, 16 and 40) were studied.Al-Fe_2O3 composite was developed by a sintering technique from the mixtures of compacted powders of Al high purity and α-Fe_2O_3 under a temperature of 700 °C for 1 hour and then slowly cooled. We have not noted the formation of thermite as foreseen by the chemical reaction due to the mixture of aluminum with hematite. The evolution of crystalline microstructures and the morphologies of surface were determined by means of X-ray diffraction, thermal analysis and optical metallography. The mechanical behavior was characterized by the tests of Vickers indentation and corrosion resistance by electrochemical tests.

1 Introduction

The composite materials with metallic matrix as Al/Fe_2O_3 can be produced by various techniques from the liquid state, the solid state, or the state vapor [1]. Zhijian Yin et al reported that Dong et al.[2] stated that the composite coatings prepared by plasma spraying's Fe_2O_3–Al self-reaction composite powders possessed multiphase metal and ceramic coexistence This significantly decreased the brittleness and increased the wear resistance of coatings even when the load was up to 490 N.
The thermite reaction between aluminium and iron oxide can be written $Fe_2O_3 + Al \rightarrow Fe + Al_2O_3 + \Delta H$ (1)ΔH : is released during reaction, ΔH can be estimated from literature [3,4,5,6] ΔH_{Al2O3} = -335 KJ/mole and heat of formation of Fe_2O_3 is –168 KJ/mole. The thermite is a mixture of metallic aluminum and oxide of another metal, generally the iron oxide. The aluminothermal reaction in which the aluminum is oxidized and the metallic oxide reduced was discovered by Hans Goldschmidt in 1893 whopatented the process in 1895.This chemical reaction generates an intense heat which can reach 2204,4 °C. Al-FeO composites can be used in aeronauticalor in electronic industry[3]. The aim of this work is to study the mechanical and physico-chemical behavior of various Al-Fe_2O_3 composites with different hematite contents ranging from 2% to 40%, as well as in the produced state treated with heat at 500 ° C. This work is a more extended version of the results presented at the 21st mechanical conference. See reference [7].

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2 Experimental

A set of samples of massive alloys, Al (base)-X wt%-Fe_2O_3) of nominal compositions X = 2, 4, 16 and 40 wt.%-Fe_2O_3) was developed by the liquid-phase sintering technique at 700 °C during1 h Fig. 1.

After appropriate metallographic operations of cutting, polishing, and chemical etching, alloys developed were characterized by the X-ray diffraction analysis XRD ($\lambda_{Cuk\alpha}$ = 0,154 nm), optical observations, and micro hardness tests by means of the light microscope and Vickers microindenter. They were equipped with a Mitutoyo HM112 microscope under a load of 150g.Electrochemical tests have been achieved in salty area (3.5%NaCl at room temperature). A SETRAM DSC apparatus has performed thermal analysis of the samples. The rate of heating-cooling loop was about 10°C/min.The homogenization heat treatments post sintering were realized for all samples at 500 °C during 1 hour.

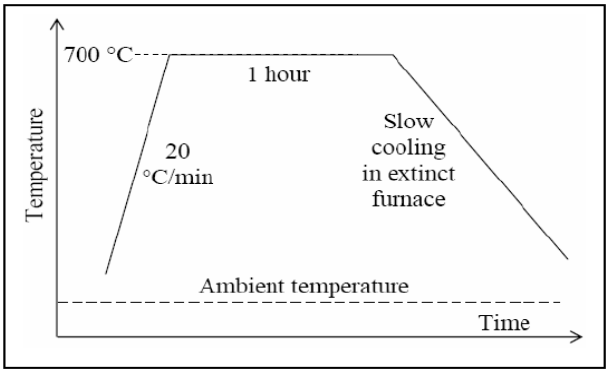

Figure 1. Heating versus time steps during the elaboration of Al-(α-Fe2O3)

3 Results and discussion

The XRD analysis shows in Fig. 2, that the line $(111)_{Al}$ appears as the most intense in the studied alloys: for Al-2 and 4 wt% (α-Fe_2O_3) alloys, the crystalline microstructures appear as the structure is predominantly solid solutions FCC Al with rhombohedral α-Fe_2O_3 phase in the FCC Al matrix, for Al-16 and 40 wt%.α-Fe_2O_3alloys, microstructures appear as being biphased a mixture of two phases with ascendancy of the matrix phase FCC Al.

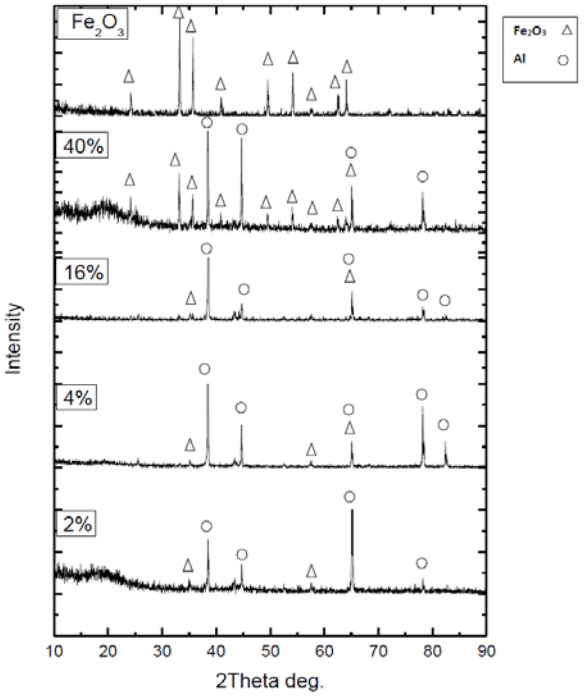

Figure 2. The XRD diffraction pattern of a set of produced Al-Fe_2O_3 composites with various Fe_2O_3 compositions

As showed by the XRD pattern and optical micrographs of the Al-(α-Fe_2O_3) alloys with 2%, 4% and 16% Fe_2O_3, as–sintered and after the heat treatment in Fig. 3 and Fig. 4a-a'/b-b' /c-c'.

Aluminium lattice constant variation with hematite content as shown in Fig. 5, for both sintered and heat-treated states of materials, is monotonically increasing from pure aluminum up to 4% Fe_2O_3 in order to decrease, then slowing down to 40 % Fe_2O_3.

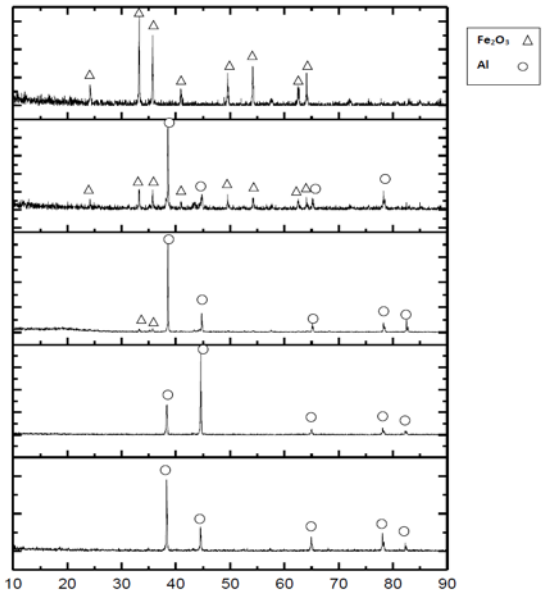

Figure 3. The XRD diffraction pattern of a set of heat-treated Al-Fe_2O_3 composites with various Fe_2O_3 compositions

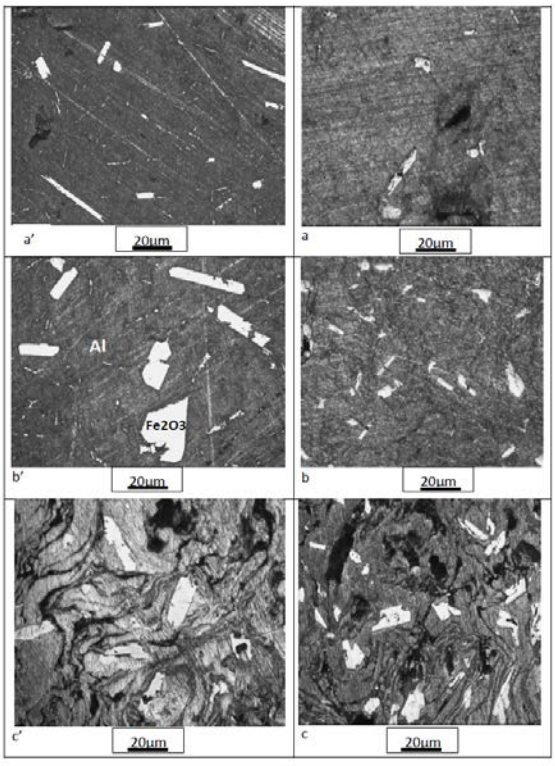

Figure 4. Optical micrographs of the alloys a) Al-2 wt%. α-Fe_2O_3, a')- Al-2 wt%. α-Fe_2O_3 heat-treated. b) 4%, b') 4% heat - treated.c) 16%, c') 16% heat-treated. Aluminium (grey) and Fe_2O_3 (white)

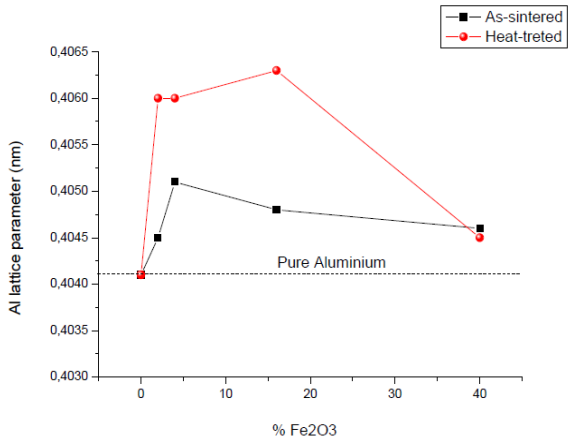

Figure 5. Aluminium lattice constant variation with Fe_2O_3 content

The situation is different for the heat-treated materials where we note a sudden increase of lattice constant up to 2% Fe_2O_3, while keeping the same value up to 4% and then increasing it up to 16%. Then it finally drops almost brutally to 40%. The thermal analysis was realized by differential scanning calorimetry on the whole composition. The range of the studied system is shown in Fig. 6 a(2%), b(4%), c(16%), d (40%) , e(pure Fe_2O_3) and allows us to notice that with the exception of pure Fe_2O_3, the other composites Al-Fe_2O_3 behave almost in the same way in heating as in cooling with a small difference in Al-40%Fe_2O_3 composite. It is a worth knowing appearance of an exothermic peak in thevicinity of 650 °C as a result of the aluminum fusion.

What can draw our attention at first sight from a view of various DSC recordings is the importance of the loop of hysteresis to the temperatures between 650 °C and 750 °C (marked by a dashed circle in Fig. 6(a,b,c,d,e) when we turn back to begin the phase of cooling .The width of this part of loop is dependent on the content in Fe_2O_3 of the considered composite material. More Fe_2O_3 that is contained in materials, wider is the loop.This phenomenon can be interpreted as if the scale of heat cleared during the reaction that took place between Al and Fe_2O_3. This phenomenon can be interpreted as the amount of heat released during the reaction between Al and Fe_2O_3 in an aluminothermic type reaction such as that which occurs when aluminum is mixed with iron [3,4].

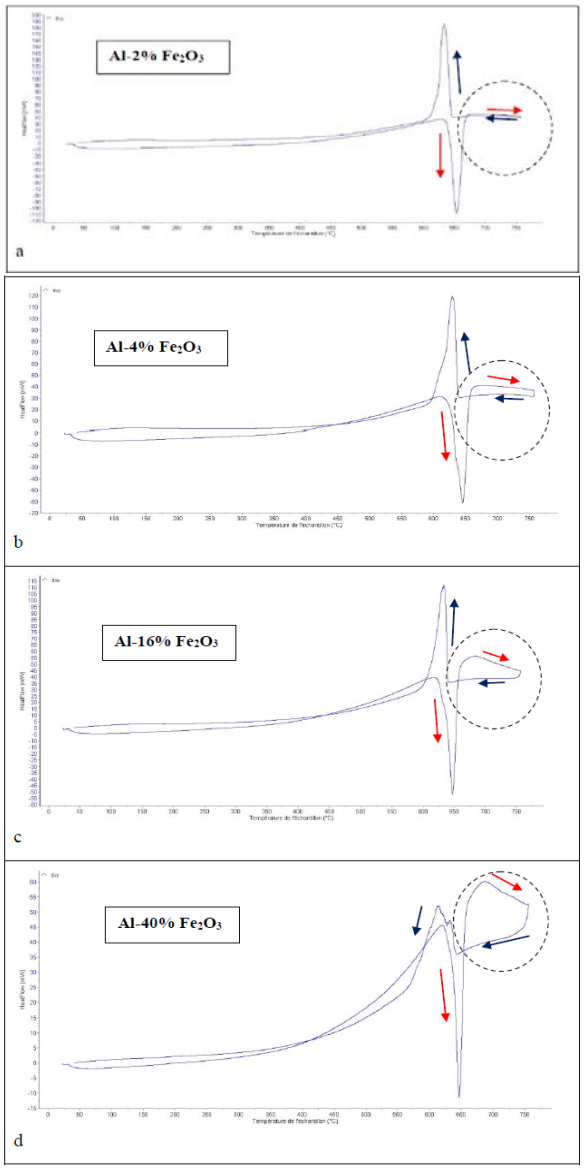

Figure 6. (a,b,c,d,e) Differential scanning calorimetry monitoring from the studied composites with various composition in heating (Red arrow) and cooling (Blue arrow)

The Vickers $Hv_{0.2}$ indentation allowed to estimate the micro hardness of alloys Al-2 and 40 wt.%(α-Fe_2O_3) where we observed the same evolution as that of Al-2 and 40 wt% (α-Al_2O_3) [8,9] or in composite materials Al/Al_2O_3 developed by technique of sintering of powders [10]. So, the Vickers microhardness, which is compared with that of a pure aluminum produced by high frequency fusion induction fusion (200 MPa) [9], reached the value of 450 MPa for the alloy of composition Al-16 and 40wt.%(α-Fe_2O_3) Fig. 7. This hardening observed in aluminum reinforced by the hematite α-Fe_2O_3 is of an essential behavior in the densification of the material by insertion of the rhombohedral phase α-Fe_2O_3 in the FCC Al matrix by the technique of elaboration by liquid-phase sintering [10,11].

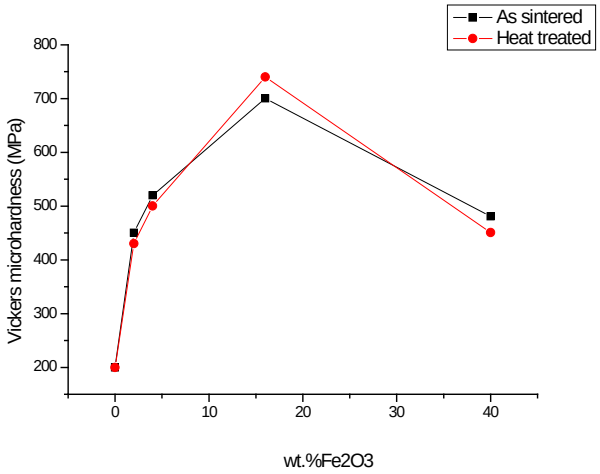

Figure 7. Microhardness evolution versus Fe_2O_3 content in Al-Fe_2O_3 composites (As-sintered and heat-treated)

The addition of Fe_2O_3 in aluminum causes an increase in hardness of the composite material in a very significant way [12].Besides, the hardness increases in a monotonous ways as well as the produced state that the heat-treated one until a maximum which corresponds to 16%Fe_2O_3 to fall to 40 %Fe_2O_3 Fig. 7. If we consider that, the hardness of this material obeys the same law, which governs that of a composite material. This means that the value of the hardness is connected to the mass fraction of phase Fe_2O_3 embedded in the aluminum matrix $H_v = H_{v.Al}.(1-v) + H_{v.Fe2O3}\ v$ with v: mass fraction of Fe_2O_3 in the material [13]. Fig. 8 shows the shape of the pyramidal indentation impact on the material surface.

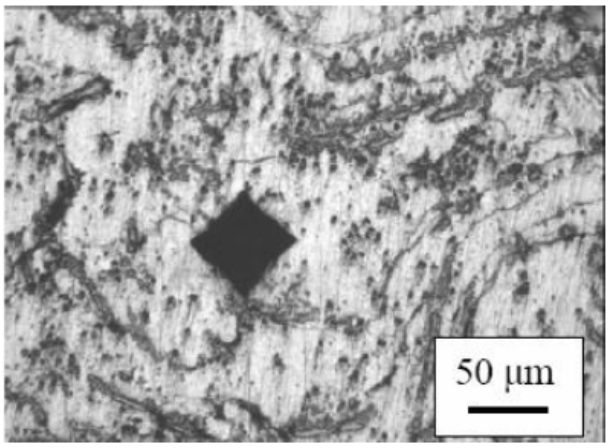

Figure 8. Shape of the pyramidal indentation on material surface

The porosity rate of the various samples taken in their raw state decreases sharply as a function of the Fe_2O_3 content incorporated in the aluminum matrix Fig. 9.In the heat treated specimen, the decrease is slower. In the case of the treated samples, the overall porosity rate is inferior to that of the raw samples, this is normal, since most of the pores have been welded by diffusion after treatment. Whereas, in the case of an Al-Al_2O_3 composite material, the amount of porosity and density in the composites increased with increasing weight fraction of the particles. [14] [15][16] [17].

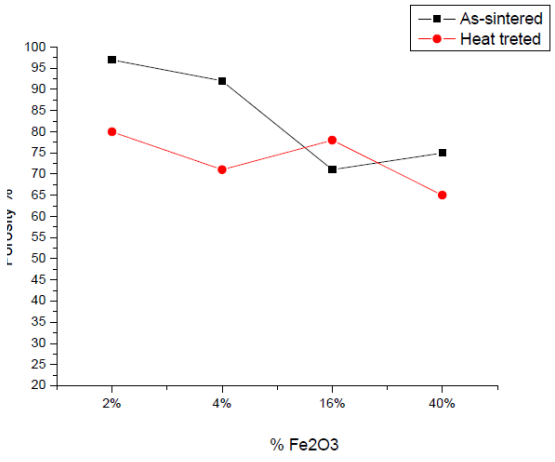

Figure 9. Porosity ratio variation versus Fe_2O_3 content (As prepared materials and heat-treated).

For the same samples treated at 500 °C, for 1 hour the porosity rate decreased between 2% and 4% and then increased to 16%, followed by a slower decay as observed in the produced state. The general tendency of decay in both cases are in the crude state of sintering and after subsequent heat treatment.

The corrosion behavior of the composite materials studied in this work is explained in terms of the variation of the corrosion rate with the Fe_2O_3 content as shown in Fig. 10, where there is a quasi-monotonic decrease in the rate of corrosion between 2% and 40% Fe_2O_3. This behavior shows that if more aluminum is charged with iron oxide its corrosion rate is very low, as is the case of Al-40% Fe_2O_3 for example.

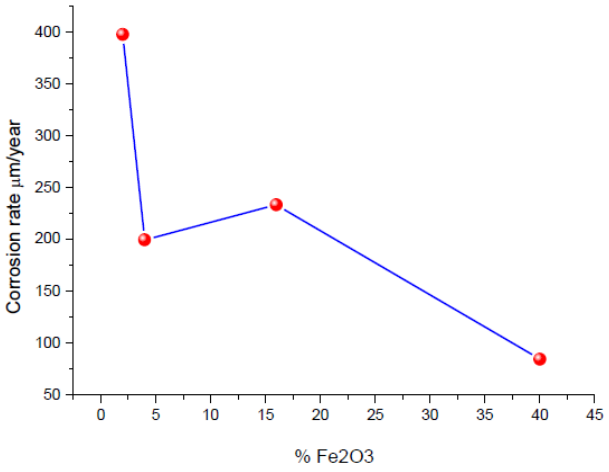

Figure 10. Corrosion rate versus Fe_2O_3 composition (2%, 4%, 16%, 40%)

The Taffel extrapolations in Fig. 11 shows a singularity displayed by the sample containing 40% Fe_2O_3 that has the best electrochemical performances due to the lowest icorr and lowest corrosion rate. This is compared to the whole range of composite materials of the same family. The decrease of the porosity caused by the additions of Fe_2O_3 powders causes a decrease on the corrosion rate [18], due to the small surface exposed to the corrosion phenomenon. This specimen composition choice could lead to promising material for use in severe medium [19], competitive with other corrosion resistant functional materials.

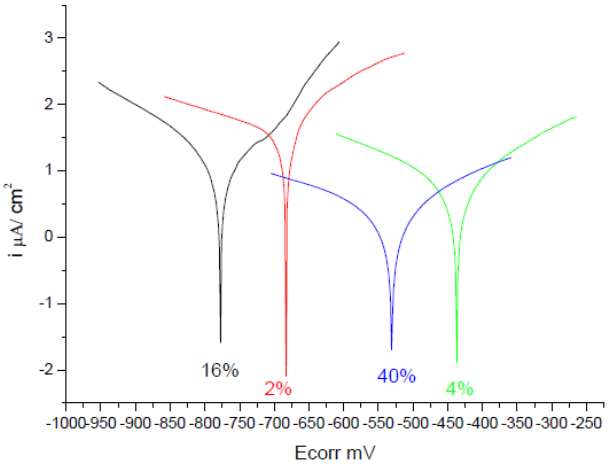

Figure 11. Taffel curves of the composite materials Al-Fe_2O_3 with various compositions.

The evolution of the corrosion potential with the content of hematite Fig. 11 reveals that the 4% of Fe_2O_3 mixture possesses the highest corrosion potential of the entire range of studied products. Especially since its value is very close to that of the pure iron, this can be explained by the fact that the reaction $Al+Fe_2O_3 \rightarrow Al_2O_3 + Fe$ did indeed take place after sintering, unlike the other three. Based on the results obtained, it can be deduced that Al_2O_3 improves the corrosion resistance because Al_2O_3 affects the anodic and cathodic reactions [20].

4 Conclusion

This study made it possible to show the influence of the addition of Fe_2O_3, according to several grades, as reinforcement in the Al-Fe_2O_3 composite material, on the physical, physico-chemical, and mechanical properties. At the end of this work, two special compositions have attracted our attention, Al-4% Fe_2O_3 and Al-40% Fe_2O_3. The corresponding composite material meets the standards for their possible use in saline atmosphere. The mechanical properties, through the tests of micro hardness and porosity measurement are also of interest for most invested compositions. The rate of porosity decreases significantly with the hematite content, unlike the other composites with Al_2O_3 reinforcement. The addition of Fe_2O_3 in the aluminum causes an increase of the hardness of the composite material in a very significant way.

5 Dedication

This paper is dedicated in the memory of Professor Mohamed Draissia.

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