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Development of a new aluminium matrix composite reinforced with iron oxide (Fe_3O_4)

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Materials

ABSTRACT

Purpose: of this paper is to develop new aluminium matrix (intermetallic) composites reinforced with iron oxide (Fe_3O_4) that will be used in aeronautical engineering or in electronic industry. Different parameters such as sintering time and temperature, reinforcement, compact pressure were evaluated. The final purpose of this project is going on to improve conductivity and magnetic permeability of this new composite.
Design/methodology/approach: In this paper, a new alternative materials "aluminium–iron oxide (Fe_3O_4, naturally as the mineral magnetite) powder composite" has been developed by using a microwave (in the laboratory scale) sintering programme with various aspect ratios, that iron oxide (Fe_3O_4) particle sizes and aluminium powders together were prepared. This paper contains partially preliminary results of our going-on research project.
Findings: Green density increased regularly depending on the compact pressure and percentage of the iron oxide (Fe_3O_4). Micro and macro porosity was not found due to very clean microwave sintering. Density after microwave sintering was higher than that of traditional sintering in an electrical oven.
Research limitations/implications: This project is going on and magnetic permeability and conductivity of this composite will be improved.
Practical implications: This composite is new and clean and thanks to the new microwave sintering basically will be used in aeronautical engineering. Microwave heating results in lower energy costs and decreased processing times for many industrial processes.
Originality/value: Originality of this paper is to use a new reinforcement in the aluminium matrix composite; Fe_3O_4-iron oxide. A new method - microwave sintering- has been carried out on this composite.
Keywords: Intermetallic composites; Aluminium- Fe3O4-iron oxide; Conductivity; Magnetic permeability; Microwave sintering

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1. Introduction

In modern industry, it is more and more enforced to develop new composites, such as high resistant, alternative materials of low density in order to realise multifunctional pieces. The powder metallurgy components are being widely used for sophisticated industrial applications. The worldwide popularity of powder metallurgy is about the ability of this technique to produce such complex shapes with exact dimensions at a very high production rate and low cost.

For this reason, it is very striking to use reinforced (Fe_3O_4-iron oxide) aluminium matrix composites in structural applications (automotive, aeronautical, etc.) due to their outstanding stiffness-to-weight and strength-to-weight ratios. These materials show good thermal conductivity and wear resistance and also low thermal expansion, all of which makes them very high, multifunctional, of light weight materials. Additionally, it is a very attractive way to add Fe_3O_4-iron oxide reinforcement to improve the magnetic permeability of this composites and by this way, good synchronization can be obtained between thermal and electrical conductivities and magnetic permeability.

In the literature, many papers have been carried out on this material, essentially a very detail study was made on strain hardening behaviour of Al-Fe powder metallurgy composites and a new constitutive relationship has been proposed for the strain hardening behaviour of Al-Fe powder composites [1-5].

In other investigations [6], results on elemental Al matrix composites, reinforced by 30 vol. % of spherical Al–Cu–Fe alloy particles and consolidated by quasi-isostatic forging, were introduced. Because of the fine (diameter<10 μm) matrix and reinforcement particle sizes that match closely and a homogenous spatial distribution, the yield strength (YS) of this model composite material was improved over the matrix properties by 220% for the commercial purity composite sample.

Remarkably, for equivalent composite material produced from higher purity metal powders, i.e., with much thinner oxide surfaces, the yield strength of the composite was improved over the matrix properties by 328%. Because these strengthening results have been so far above previous observations, typically <100% [1-6].

These results suggested also that the selection of potential reinforcement phases for particulate reinforced aluminium materials should be extended from refractory ceramics to include, e.g., intermetallic compounds that also can form a strong bond with the Al matrix by high temperature solid-state sintering. Additionally, the results clearly indicated that PRA composites made of powders with a thinner oxide surface can achieve significant improvements in tensile properties over the same composites made of commercial powders with residual impurities and thicker oxide surfaces [6].

On the other hand, oxides of iron are a proven candidate in self-sustaining aluminothermic reaction as in thermite welding [7]. Fe_3O_4 is considered as suitable for its lower cost and high free energy of reaction. Such a reaction, besides improving wetability between oxide and aluminium matrix, would also provide extra energy for the process [7-8].

Mechanical alloying is a useful powder processing technique that can produce a variety of equilibrium and non-equilibrium alloy phases. The advantage of this process technology is that the powder can be produced in large quantities and the processing parameters can be easily controlled, thus it is a suitable method for commercial applications [14-34].

In general, some magnetic properties can be improved when the grain size is reduced to the nanoscale, while the presence of stresses and defects introduced by mechanical alloying impairs the magnetic property; the overall magnetic property is a competition between decrease in grain size and increase in strain. Even though the strain can be removed by heat treatment of the as-milled powder, the grains grow during a heat treatment.

In the light of the literature survey [1-34], a new study of this type of metal matrix composites was undertaken for this report that there is a lack in the literature according to our knowledge. In this paper, a new alternative materials “aluminium–iron oxide (Fe_3O_4, naturally as the mineral magnetite) powder composite” has been developed by using a microwave (in the laboratory scale) sintering programme with various aspect ratios, that iron oxide (Fe_3O_4) particle sizes and aluminium powders together were prepared. This paper contains partially preliminary results of our going-on research project.

2. Experimental details

2.1. Materials

In this study, pure aluminium of 99.7% purity was used as the base material (Merck Co, France), grain size was reported as 2 μm. Aluminium and iron powders of the description, stated in Table 1 were procured and analysed for its purity.

The purity level of iron oxide powders (Fe_3O_4) was found to be 99.62%. The compacts were prepared from aluminium and iron powders with various aspect ratios varying from 0.25to 0.85 as given in Table 1. Grain size of iron oxide powders (Fe_3O_4) was reported as 2 μm.

Table 1.
Measured density for the specimens after sintering in different aspect ratio

Density, kg/m3	Aspect ratio
2710	0.45
2710	0.56
2720	0.83
2728	0.76
2730	0.34
2723	0.54
2906	0.22
2987	0.35
3054	0.63

As the starting materials for the processing of the composites were arranged according the percentage of Fe_3O_4 and coded as Al-Fe2, 4, 6 9, etc. in various wt% powders (hereafter denoted as AF2, AF4, AF-MW, AF6 and AF9).

All of the compositions with different molar ratios (with Fe_3O_4 iron oxide contents varying from 1 to 25%) have been blended homogeneously in a ball mill for one hour with natural ethanol together.

2.2. The specimens compacting

The compacts geometry was prepared from aluminium and iron oxide (Fe_3O_4) powders Then, blended powders were compacted by cold isostatic pressing (CIP) with a green compact pressure of 275 MPa, intending to produce an initial green density ranging from 85% to 95%.

The aspect ratio of this geometry was 0.85. The other series were prepared with various aspect ratios at the green compact pressure range of 200-275 MPa in order to observe the effect of the compact pressure on the evolution of the density. Some of these specimens were shown in Figure 1.

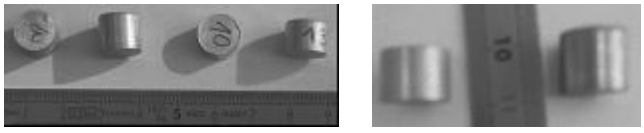


Fig. 1. Specimens prepared with different aspect ratios

For each composition, 3-4 specimens were prepared for microstructural analysis. All of the values measured here are the mean values from measurements taken on the 3-4 specimens.

2.3. Microwave - sintering

The application of microwave energy for processing of various materials such as ceramics, metals and composites offers several advantages over conventional heating methods. Microwave heating results in lower energy costs and decreased processing times for many industrial processes.

These advantages include unique microstructure and properties, improved product yield, energy savings, reduction in manufacturing cost and synthesis of new materials.

Firstly, some of the specimens were sintered in an electrical - conventional furnace (High Temperature Furnace) in order to compare the microstructural evolution regarding to the sintered specimens in microwave oven. In this project, the primary aim, however is to use a microwave oven of house type in laboratory conditions. For this reason, a house type (2.45 GHz) microwave oven was used by converting some of the the microwave oven conditions.

First of all, a temperature measurement system was adapted to the microwave by using a special thermocouples covered with safety insulating cables to prevent the electrical contact of the thermocouples when microwave is in operation. The accuracy of temperature measurement including this device was determined to be within -10°C of the measured temperature.

The thermocouples were inserted inside of the alumina crucible that was very close (2 -3 mm) to the specimens.

The input power for microwave heating was adjusted manually to control the sintering temperature and heating rate. The samples were placed in a crucible surrounded by a special insulating safe box made of alumino-silicate. Figure 2 shows the design of this safe box prepared for microwave sintering. Very fine SiC powder of 1-3 µm particle size was used as a microwave absorber. The SiC powder was placed inside of another alumina crucible

After the green compacting, the specimens have been sintered in a microwave oven using two power levels; 500W and 750W for each short period of time. Sintering temperature was measured between 500 - 550°C. When the sintering programme was finished, the specimens had been cooled inside the oven.

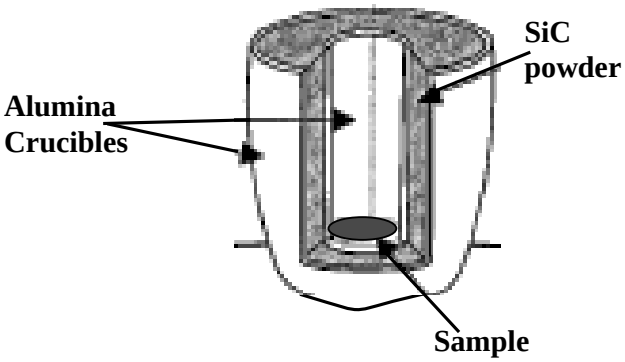


Fig. 2. Design of the safe box prepared for microwave sintering and using a house type microwave in laboratory conditions

Compared to the conventional sintering, there was a slight increase in density of the sintered specimens using microwave conditions. Since the microwave sintering took much shorter time, densification rate in the microwave sintering process might be considered to be higher than while conventional sintering.

To see the effect of sintering time on densification, samples were sintered for 10, 20, 30 and 45 min. Above 20 min of sintering; there is not much change in densification, especially when the green density is high.

2.4. Microstructural analysis and other tests

For the characterisation of the sintered specimens, microstructural analysis was evaluated using an optical microscope (Zeiss), a Joel-Oxford S-4200 scanning electron microscope (SEM). Energy-dispersive spectroscopy (EDS) and X-ray diffraction were utilized to determine the composition of the matrix and oxide phases.

Image analysis techniques were used to determine the volume fraction of iron oxide dispersion for each composition. And also, electrical conductivity of the sintered specimens was measured by means of “Agilent 4284A” with a precision - LCR Meter; Measures → Z-θ, Tension → 1 V and frequency → 20 Hz.

And magnetic permeability of the sintered specimens were measured by means "Gaussmeter-Wuntronic-DC and AC Field Measurement to 20 kHz" that contains a Computer Controlled Measuring Equipment (CCME) for the determination of magnetic characteristics of hard- and soft magnetic materials (Hysteresis-graphs) in the laboratory conditions.

Finally Microhardness test were made for all of the specimens with $HV_{0,025}$ measurement.

All of the densities of the specimens were determined using the Archimedes' principle.

3. Results and discussion

At the first stage of the out-going project, the central point is to characterize intermetallic particles from Al–Fe system formed in an aluminium matrix composite obtained by a powder metallurgy method using a microwave sintering process conditions. Figure 3 shows an Al-Fe phase diagram computed for the Al-Al_3Fe [1]. As it is seen from this diagram, the formation of solid Al_3Fe (fcc) occurs at the sintering temperature of the composite proposed in this project. It means that a simple role exchanged between Fe and Al occurs originating the Fe-Al dispersion. Deux different Al-Fe compositions were given in the Figure 4 as typical optical microscopy image showing the Al_3Fe particles position. Herewith, a good distribution of the reinforcements in the matrix was confirmed maintaining the same experimental conditions for all other mixtures.

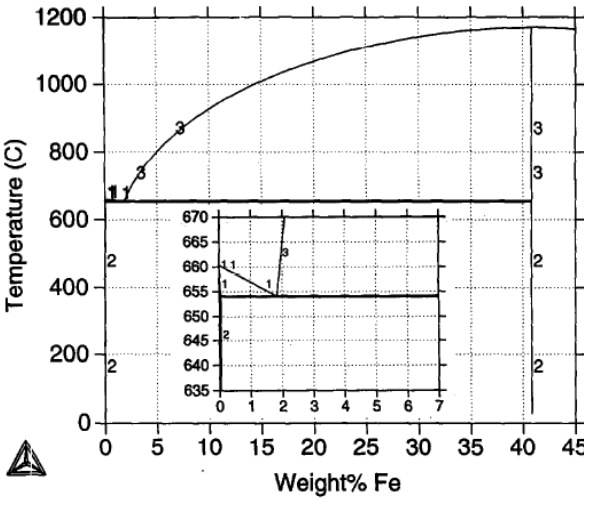
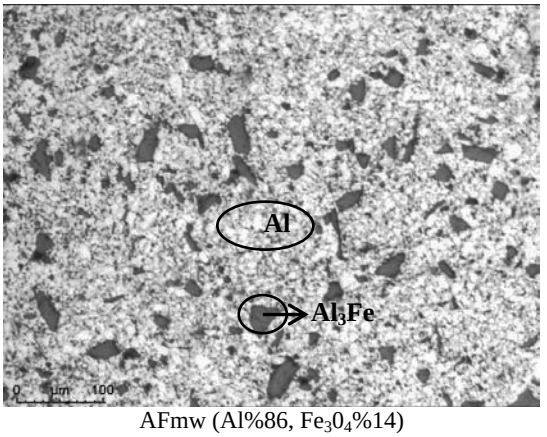


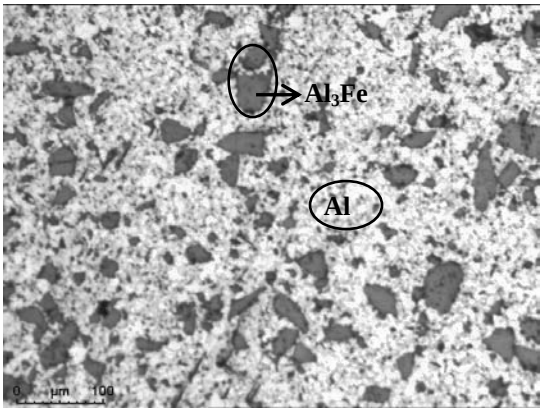
Fig. 3. Al-Fe phase diagram computed for the Al-Al_3Fe [14] equilibrium 1→fcc liquid, 2→Al_3Fe fcc-solid, 3→Al_3Fe liquid

As shown in these micrographs of Figure 4, the reinforcement particles are spherical and rectangular in shape and distributed quite uniformly in all samples. Some local clusters still can be found in all samples, especially in higher vol. % loading samples.

However, more details for distribution and position of the reinforcement can be well observed from the SEM images as shown in the Figure 5. All the specimens presented in this Figure have been produced at the same conditions; at the same green compact pressure-250 MPa and sintering of the pressed specimens at the same temperature of 550°C in microwave oven. The micrographs were taken from the middle region of the composite sections. Distribution of the particles were uniform but many of the specimen had higher Fe_3O_4 concentration levels.



AFmw (Al%86, Fe_30_4%14)

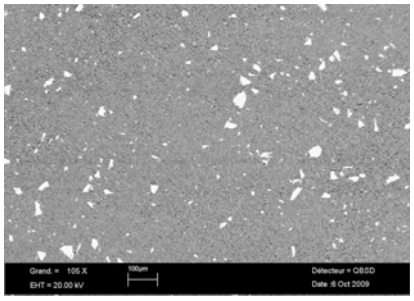


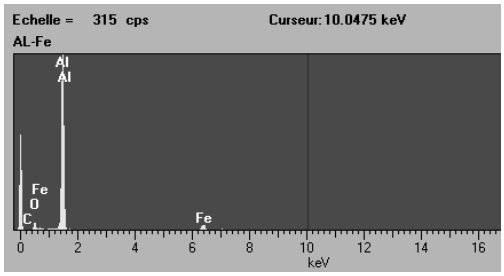
AF6-1 (Al%82, Fe_30_4%18)

Fig. 4. Typical optical microscopy image obtained from two different compositions showing the Al_3Fe particle position

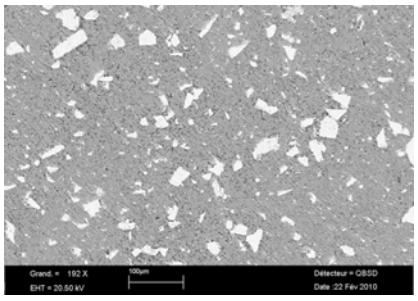
X-ray diffraction measurements of the consolidated composites exposed elemental Al matrix phase peaks, but the second quasi-crystal phase peaks, in the reinforcement particulates. However, these intermetallic particles were only the formation of Al_3Fe that is stable when the sintering temperature attained at about 550°C [6-16].

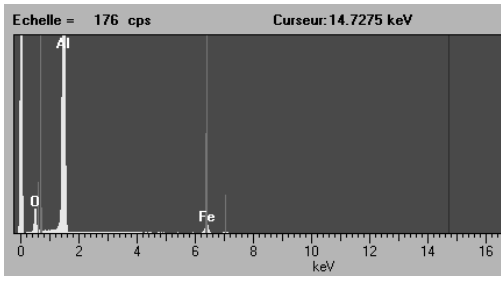
In fact, Fe_3O_4 may react with Al phase to form this type of formation during sintering at 550°C, it is reasonable to expect that Al atoms may diffuse into the Fe quasi-crystal particles and may react to form this phase. During the observation of these specimens in the SEM, we have carried EDS analysis on the surface of the agglomerated Al_3Fe particles, and Al atoms found in these particles varies in a very large gap from 2 to 15.



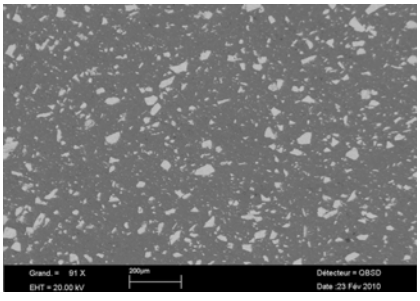


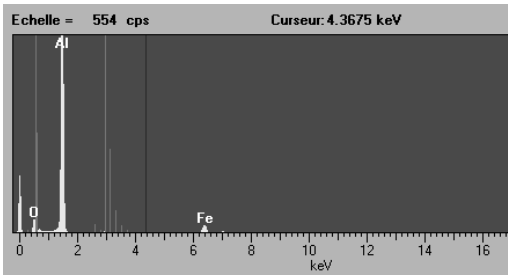
AF2 (Al%98, Fe_3O_4%2)



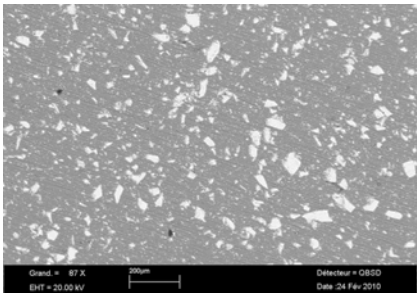


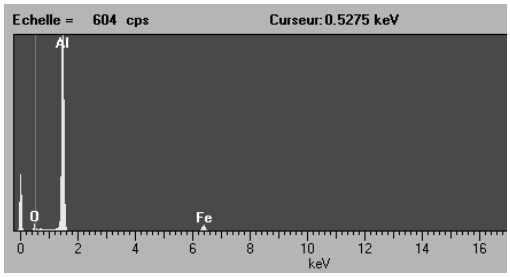
AF4 (Al%92, Fe_3O_4%8)



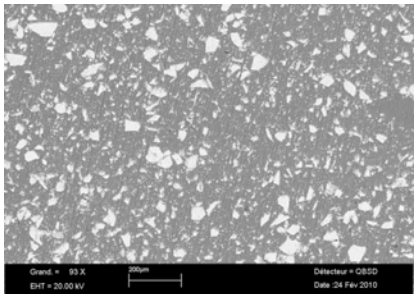


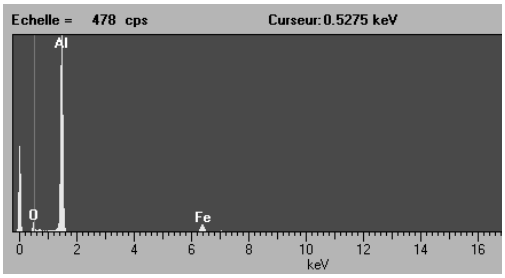
AF-MW (Al%86, Fe_3O_4%14)





AF6 (Al%82, Fe_3O_4%18)





AF9 (Al%77, Fe_3O_4%23)

Fig. 5. SEM micrographs of microstructures of Al-Fe_3O_4 composite samples AF2, AF4, AF-MW, AF6 and AF9

Figure 6 displays evolution of density after sintering depending on the green compact pressure. These evolutions have done only one aspect ratio (0.55) and only one composition (Fe_3O_4 8wt %). It is observed from this Figure that density after sintering increased with the increase of green compact pressure.

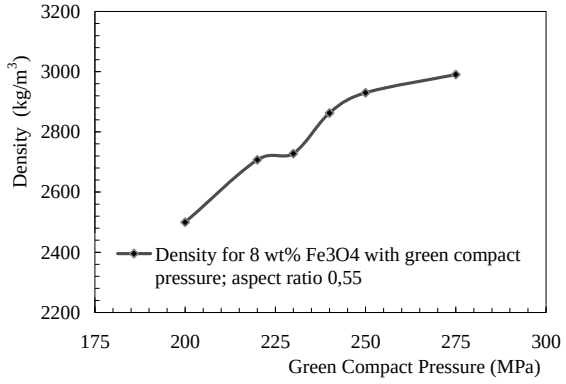


Fig. 6. Evolution of Density after sintering depending on the green compact pressure (aspect ratio→0.55; Fe_3O_4→8wt %)

These results are reliable with the former studies in the literature. In the frame of this project, evolution of the density depending of the other parameters sintering time and sintering temperature (not presented here) displayed that there were no significant effect after 45 minutes for sintering time and 530°C for sintering temperature. In fact, all of the density measurements of the samples with different aspect ratios as given in Table 1 show that all of the samples are essentially fully dense.

In the same way, development of the density after sintering has been evaluated as a function of the percentage of reinforcement (Fe_3O_4-iron oxide). This evolution was displayed in Figure 7. As it is seen from this Figure, density after sintering increased very sharply after the value of 15wt % iron oxide (Fe_3O_4). These values should be optimised with other properties such as magnetic permeability, conductivity, hardness etc. Figure 8 displays evolution of the hardness values of the samples depending on the percentage of the reinforcements in the matrix. As for the other parameters related to the magnetic permeability and density, it seems that it is suitable to optimise the percentage of the reinforcement particulates. The results obtained in this stage of the current research project show that the optimised value of the iron oxide (Fe_3O_4) is the level of 8%.

The second stage of the pending project is to improve the magnetic and structural properties of this composite. For this case, two important magnetic properties should be clarified; coercivity (H_c) and saturation magnetization (M_s).

The coercivity is often seen as an important parameter if low losses are to be achieved and affected by the most types of defects. This includes dislocations, grain boundaries, precipitates and non-magnetic particle distribution. This magnetic property is an important factor for identification of soft magnetic behaviour [2, 15-17]. The high amount of the coercivity is due to internal micro-strain, impurities, pores and defects which are introduced during milling. To reduce the coercivity, the values of all these factors should be low.

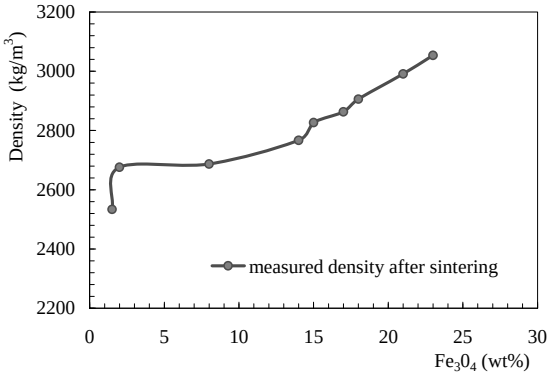


Fig. 7. Evolution of density after sintering depending on the iron oxide (Fe_3O_4)

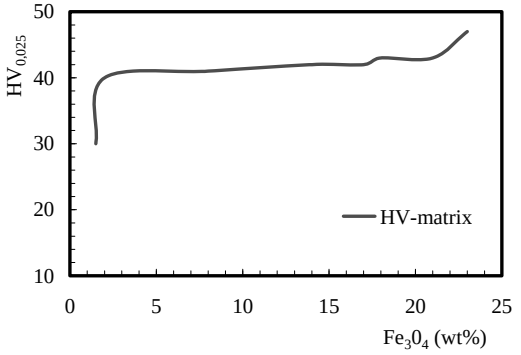


Fig. 8. Evolution of hardness after sintering depending on the percentage of the reinforcements in the matrix

The saturation magnetization is generally regarded as independent for the microstructure and strongly depends on the chemical composition. Ms which is the other important parameter from a magnetic point of view increases when milling time is increased. This could be certainly attributed to the reduction in magneto-crystalline anisotropy due to the grain refinement, which leads to an easier rotation of the domain walls [2, 15-19].

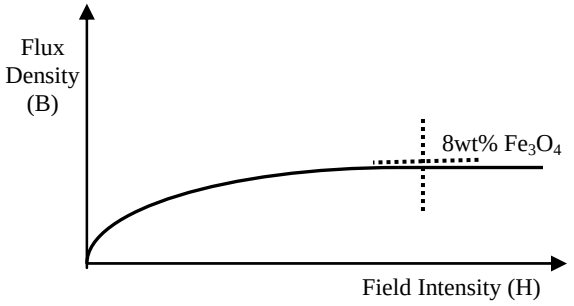


Fig. 9. Schematic presentation of the composite AF4 (Al wt 92%, Fe_3O_4 8wt %)

In the present paper,, we will give only two measurements carried out on just one composition (8wt % iron oxide, Fe_3O_4) by

an external specialised laboratory as detailed in the section of the experimental conditions (Schematic presentation was given in the Figure 9). Both of the measurements have been carried out on the 4 specimens and means values are given as follows just for AF4 (Al wt 92%, Fe_30_4 8wt %):

- AF4 Fe_30_4 8wt %)
- Magnetic permeability μ (H m^{-1}) → 7.5 x 10^{-4}
- Electrical conductivity (S·m^{-1}) → 15.62 x 10^6

At the second stage, the current project will be focused on the detail analysis of the magnetic properties.

4. Conclusions

In this paper, a new alternative materials "aluminium–iron oxide (Fe_3O_4, naturally as the mineral magnetite) powder composite" has been developed by using a microwave (in the laboratory scale) sintering programme with various aspect ratios, that iron oxide (Fe_3O_4) particle sizes and aluminium powders together were prepared. This paper contains partially preliminary results of our pending research project. This new aluminium matrix (intermetallic) composite reinforced with iron oxide (Fe_3O_4) will be used essentially in aeronautical engineering or in electronic industry.

First of all, certain parameters such as sintering time and sintering temperature, percentage of the reinforcement, green compact pressure, etc. were evaluated. The final purpose of this pending project will be focused on the improvement of the conductivity and magnetic permeability of this new composite.

Our experimental results showed that the density after sintering increased regularly depended on the compact pressure and percentage of the iron oxide (Fe_3O_4). There was not found micro and macro porosity due to very clean microwave sintering. Density after microwave sintering was higher than that of traditional sintering in an electrical oven.

This composite is new and clean and thanks to the new microwave sintering and basically will be used in aeronautical engineering. Microwave heating results in lower energy costs and decreased processing times for many industrial processes.

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