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Original Article

Technical and environmental evaluation of a new high performance material based on magnesium alloy reinforced with submicrometre-sized TiC particles to develop automotive lightweight components and make transport sector more sustainable

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ABSTRACT

This study evaluated the use of submicrometre-sized particles based on titanium carbide from both technical and environmental points of view. The objective was to improve the mechanical properties of the magnesium alloy intended for use in the automotive component industry. To this end, an Al/TiC master compound containing 60 wt.% of TiC was produced through a self-propagating, high-temperature synthesis process and embedded in a magnesium alloy by a mechanical stirring method. The life cycle assessment methodology was then used to evaluate the environmental impact of the manufacturing of the magnesium alloy reinforced with submicrometre-sized particles. X-ray diffraction and scanning electron microscopy techniques revealed the nature and purity of the TiC present in the material and revealed particle sizes below submicrometre range (300–500 nm). The incorporation of TiC particles into the magnesium alloy resulted in improvements in yield stress and ultimate tensile strength of more than 10% and 18%, respectively, and increases in ductility values by 30%. Finally, the results indicated that the submicrometre particle production had a low environmental impact compared with the total impact associated with manufacturing

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Abbreviations: CMA, Chinese magnesium association; CTE, coefficient of thermal expansion; EC, European commission; EU, European union; GHG, greenhouse gas; LCA, life cycle assessment; LCI, life cycle inventory; SEM, scanning electron microscopy; SHS, self-propagating high-temperature synthesis; XRD, x-ray diffraction.

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the magnesium alloy reinforced with submicrometre-sized particles; the greatest environmental burden was attributed to the magnesium production stage. However, this impact is offset in the use phase of the vehicle, providing approximately 28,000 km of mileage for a car.

1. Introduction

The transport sector is one of the most polluting sectors in Europe. According to the European Commission [1], this sector releases more than 50% of the total NOx emissions, approximately 60% of CO emissions, and 25% of total energy-related CO_2 emissions. Considering the latter value, 80% of CO_2 emissions come specifically from road transport. Passenger cars provide the largest contribution, approximately 45% of CO_2 emissions. Accounting for this, one of the main priorities of the European policies is to reduce Greenhouse Gas (GHG) emissions by 60% compared to the 1990 levels [2]. The current European strategy recognises the positive results of certain measures already taken at the European Union (EU) level, but has underlined that further progress is required in research and technological development, in particular to reduce CO_2 emissions [3].

Several strategies have since been launched. Some improvements are being achieved in the performance of conventional engines, for example, using variable geometries of the valve lift and the throat diameter [4], introducing selective catalysts to reduce emissions [5], or adapting the engine for using alternative fuels or blends [6,7]. Additionally, alternative technologies to the conventional engines with a lower emission ratio are being developed, such as electric vehicles, which may be self-contained with a generator to convert fuel to electricity (hydrogen fuel cells) [8,9] or a battery that may be powered through a collector system by electricity from off-vehicle sources [10,11].

Finally, a current strategy is to improve car design by using lightweight materials to produce the car body and its components, decreasing the total weight and thus, the fuel consumption and associated emissions [12,13]. The most used materials in the automotive sector are steel and aluminium [14]. However, in recent years, there have been attempts in the automotive sector to substitute these materials with lighter ones, such as composite materials based on glass fibres [15], polymers [16], and magnesium [17]. The latter is the most promising material for structural components as it is the lightest structural metal.

The density of magnesium is around 33% lower than that of aluminium and approximately a fifth that of steel. Therefore, there is an opportunity to save fuel by using this in vehicle components and, consequently, achieve the required CO_2 emission reduction. Nevertheless, it is also important to evaluate the mechanical properties resulting from the utilisation of magnesium alloys in car parts and to ensure that they can accomplish the technical requirements of the automotive sector.

As reported by Gupta and Wong [18], although magnesium alloy components can be obtained from cast processes, these can exhibit disadvantages with regards to elasticity, ductility, and resistance properties. Although some surface protection materials such as fibrous szaibelyite [19] or other stable super-hydrophobic surfaces with pinecone-like hierarchical structures [20] are being developed to avoid corrosion, recent trends are focused on the development of new magnesium alloys based on materials including microscale reinforcements to overcome their mechanical and physical limitations.

Some studies have shown that magnesium properties are improved by the incorporation of submicrometre reinforcements. Oxides, nitrides, borides or metal carbides [18] are considered as some of the most promising reinforcements; TiC shows particular promise [21,22]. The type of synthesis method can also affect the final properties [23]. Therefore, the preparation of these novel materials generates the necessity to analyse their final mechanical and physical properties. Nevertheless, studies surrounding magnesium-based materials including submicrometre-sized reinforcements for the transport sector are very scarce and, according to the author's knowledge, there is no literature data simultaneously including a technical and environmental evaluation of the materials and production processes [24,25]. The environmental analysis conducted in this paper focused on the life cycle of an exemplary component to be used in a gasoline passenger vehicle. The component was fabricated using a magnesium alloy reinforced with submicrometre-scale particles of the Al/TiC master compound containing 60 wt.% of TiC. To this end, life cycle assessment (LCA) was the methodology applied, which is regulated under the ISO 14040 standard [26]. The LCA methodology allows the assessment of the potential impacts associated with raw material acquisition, via the production, use phases, and waste management involved throughout the life cycle of the product [27,28].

As mentioned before, an evaluation of the environmental impacts associated with the manufacturing of vehicle components using the magnesium alloy reinforced with submicrometre-scale particles was performed. This assessment was carried out through a LCA) methodology, which is useful for assessing impacts along the life cycle of a product, which broadens the scope of the environmental analysis [29]. The RECIPE method was used to classify the environmental impacts into the most representative midpoint categories of the entire manufacturing process and the results were computed using the SIMAPRO v.8 software. These are midpoint indicators evaluated using the RECIPE method. Midpoint indicators are considered to be points in the cause–effect chain (environmental mechanism) of a particular impact category somewhere between the stressor and endpoints. For such midpoints, the characterisation factors can therefore be calculated to reflect the relative importance of an emission or extraction in a life cycle inventory (LCI) by environmental metrics [27]. To develop the LCA, the production processes of both aluminium and magnesium have been considered. Bauxite, aluminium hydrate, and aluminium oxide are the main raw materials consumed in aluminium production, with bauxite as the main resource (up to 85%) [30]. An overview of the typical life cycle of an aluminium process has been reported by the Environmental Profile Report for the European Aluminium Industry [31]. Based on the scheme to produce aluminium proposed by that report and additional specialised literature, as well as articles and reports, significant variables were collected to create a complete database for environmental modelling.

The majority of the world's magnesium is produced in China and the main process used is the Pidgeon process, which has improved in efficiency over the past few years [32]. This process has been well characterised by Ehrenberger, who performed data collection based on production statistics and fuel consumption by the Chinese Magnesium Association (CMA) provided by magnesium producers [33]. Due to the fact that up-to-date and reliable data and results for magnesium production are provided, the life cycle inventory of magnesium production by the Pidgeon process is accounted for from Ehrenberger's work to create a complete LCI for the environmental modelling of this study.

Moreover, another scenario for primary magnesium production addressing the typical process carried out by a Norwegian producer has been considered. This was the world's largest magnesium producer in the 1990s. Although this facility in Porsgrunn (Norway) was closed in early 2000, this is the most representative plant for the production of primary magnesium in Europe.

Here, magnesium is produced from seawater and dolomite. The process starts by producing $Mg(OH)_2$ from calcination of dolomite by mixing with seawater followed by the precipitation and filtration of $Mg(OH)_2$. The filter cake from the latter process then undergoes calcination to produce MgO. Chlorination of MgO allows $MgCl_2$ to be obtained, which is electrolysed and eventually ingots are produced in the foundry.

Thus, the LCI for magnesium production was based on the environmental reports of Norks Hydro for the year 1998, contained in the database of the SimaPro software version 8.0 (not shown here due to confidentiality issues).

Finally, there is one other significant impact factor in the processes for magnesium alloy production. Magnesium alloy production needs a cover gas to prevent degradation from contact with the surrounding atmosphere. Two gases are usually used, SF_6 and SO_2. The Chinese Pidgeon process uses sulphur or fluxes containing small amounts of sulphur to prevent the magnesium melt from burning [34]. However, SF_6 contributes a significant environmental burden in terms of CO_2 eq., as this gas is considered a GHG. Therefore, the process such as that in the Norwegian plant, assumed in the environmental model of this study, considers an alternative gas cover, namely R134 gas, which is considered an environmentally-friendly protection gas with reference to cast magnesium.

Thus, this study aims to fill part of this information gap by a technical and environmental evaluation of submicrometre-sized particles based on TiC as an approach for improving the mechanical properties of magnesium alloy to produce automotive components. An Al/TiC master compound containing 60 wt.% of TiC was produced through a self-propagating high-temperature synthesis (SHS) process and embedded in a magnesium alloy by a mechanical stirring method. The technical evaluation was conducted by analysing the most representative physical and mechanical properties. To this end, X-ray diffraction (XRD) and scanning electron microscopy (SEM) techniques were used to investigate the nature and purity of the TiC particulates in the material and their sizes. The mechanical properties, namely yield stress, tensile strength, and ductility, were tested following the ASTM E8/E8M-13a standard.

2. Experimental techniques and methodology for the environmental evaluation

2.1. *Material preparation*

For the preparation of the magnesium composite reinforced with TiC particulates, an Al/TiC master compound containing pure aluminium and TiC particles was prepared and added to the molten magnesium alloy. The master compound was prepared by an SHS reaction of the Al-Ti-C system.

To prepare the master compound, the following materials were used: aluminium powder (supplied by Pometon España S.A. with a purity of 99.5% and average particle size lower than 200 μm), titanium powder (with a composition of Ti 99.7%, C 0.1% max., and Fe 0.2% max. provided by William Rowland Ltd. with an average particle size lower than 25 μm) and black carbon powder (with a purity of 99.5%, average particle size less than 1 μm provided by Degussa). Subsequently, in order to prepare the magnesium matrix composite, this master compound was added in powder form to a molten AM60 magnesium alloy. This alloy was supplied by Dead Sea Magnesium in Israel in ingot shape, containing a wt.% of 6.0 Al, 0.36 Mn, 0.1 Zn, and Mg (Balance).

Detail description about the preparation of the Al/TiC master compound and magnesium alloy reinforced with submicrometre-sized particles, mechanical property tests, and material characterisation are given below.

2.1.1. *Al/TiC master alloy preparation*

Titanium and carbon powders were mixed in a molar ratio of Ti/C = 1.0 and 40 wt.% of Al powder was added to this stoichiometric mixture. This was then dry mixed for 4 h using a ball mill, before being uniaxially cold pressed at a pressure of 100 MPa using a hydraulic press and a stainless steel die. A cylindrical compact with approximately 60% theoretical density was obtained. To initiate the SHS reaction, the compact was locally heated using a current of 16 V for 5 s. Submicrometre TiC particulates with a size range of 300–500 nm were formed in an aluminium matrix through an SHS reaction.

2.1.2. *Preparation of AM60 samples reinforced with submicrometre-scale particles*

For the preparation of the particle-reinforced magnesium composite, a stir casting method was employed. This method consists of mechanically stirring the molten alloy until a vortex is generated, into which the reinforcing particles are subsequently added. The master compound compact was first ball-milled for 8 h in order to reduce its size. Approximately 1700 g of AM60 alloy were then introduced in a silicon carbide crucible and heated in a high-frequency induction furnace. A k-type thermocouple was introduced in the crucible in order to measure the temperature of the molten alloy and stabilise it at approximately 700 °C for the stirring process. Once completely liquid, and after the dross layer generated in the surface was removed, a graphite stirrer was introduced inside the crucible, very close to the bottom surface of the crucible. The molten alloy was stirred at 360 rpm and the master compound powder was manually added into the vortex. The stirring was then maintained for 10 min. Finally, the stirrer was stopped and removed from the crucible and the molten material was cast in an iron mould. Once completely solid, the cast part was demoulded in order to be characterised.

2.2. *Characterisation of magnesium alloy reinforced with submicrometre-sized particles*

2.2.1. *Physical properties*

XRD and SEM were used to carry out the metallographic analysis of the cast samples. A small sample was cut from each cast part and the surface of the sample was prepared by grinding through grit papers of various sizes followed by polishing with diamond paste.

The surface morphology was investigated by using SEM, model JEOL JEM 5910 LV, equipped with an energy-dispersive X-ray analyser (EDX) to make a semi-quantitative analysis of the composition of the different phases of the sample.

The XRD pattern was recorded on a Bruker D8 ADVANCE with Cu Kα radiation and a step size of 0.02°. The Diffract plus EVA software was used to identify the crystallographic phases present.

2.2.2. *Mechanical properties*

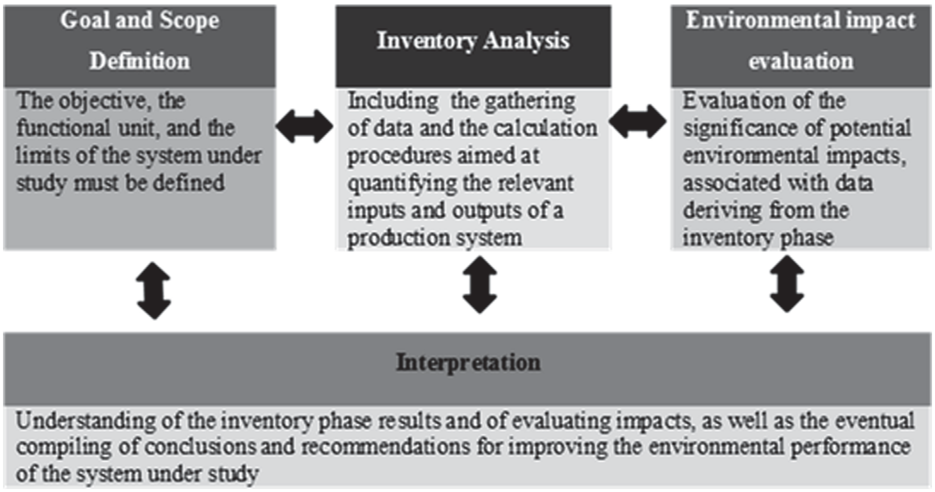
From the cast parts, tensile specimens were machined in order to be tensile tested at room temperature following the ASTM E8/E8M-13a standard; for these tests, an INSTRON machine was used. The specimen was mounted in the machine and tested at a crosshead speed of 1 mm/min. The strength and elongation were continuously recorded. Six specimens were tested for both the TiC-reinforced magnesium alloy and the unreinforced alloy.

2.3. *Environmental analysis of an automotive component based on manufacturing of magnesium alloy reinforced with submicrometre-sized particles*

LCA methodology considers four main phases to provide guidance for greater consistency and quality assurance, which are briefly outlined in Fig. 1.

2.3.1. *Scope of the environmental analysis and functional unit*

In this study, which concerns vehicle components, the identification of a lightweight material that exhibits improved mechanical properties is a feasible goal. Firstly, the evaluation was performed by the quantification of the environmental impacts of two steps, alloy production and the die casting



Goal and Scope Definition

The objective, the functional unit, and the limits of the system under study must be defined

Inventory Analysis

Including the gathering of data and the calculation procedures aimed at quantifying the relevant inputs and outputs of a production system

Environmental impact evaluation

Evaluation of the significance of potential environmental impacts, associated with data deriving from the inventory phase

Interpretation

Understanding of the inventory phase results and of evaluating impacts, as well as the eventual compiling of conclusions and recommendations for improving the environmental performance of the system under study

Fig. 1 – Main phases of an LCA study.

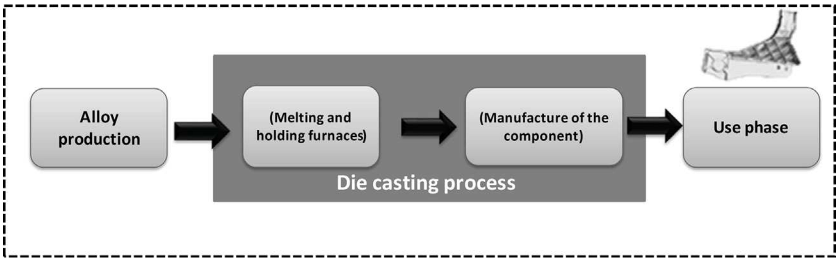


Fig. 2 – Overview of magnesium life cycle for manufacturing transport application.

Table 1 – Environmental impact indicators studied.

Impact category	Unit
Climate change	kg CO_2 eq.
Ozone depletion	kg CFC-11 eq.
Terrestrial acidification	kg SO_2eq
Freshwater eutrophication	kg P eq.
Marine eutrophication	kg N eq.
Human toxicity	kg 1,4-DB eq.
Photochemical oxidant formation	kg NMVOC
Particulate matter formation	kg PM10 eq.
Terrestrial ecotoxicity	kg 1,4-DB eq.
Freshwater ecotoxicity	kg 1,4-DB eq.
Marine ecotoxicity	kg 1,4-DB eq.
Ionising radiation	kBq U235 eq.
Agricultural land occupation	m^2a
Urban land occupation	m^2a
Natural land transformation	m^2
Water depletion	m^3
Fossil depletion	kg oil eq.

process. Secondly, the use phase was also considered calculating the CO_2 equivalent (CO_2 eq.) saving during the lifetime of a car using a light component (see Fig. 2).

The methodological framework of this study includes the impact midpoints indicators described in Table 1. The functional unit is declared as one final part of the component evaluated (generic body-joint component).

2.3.2. *System description and boundaries*

In order to include relevant effects during the life cycle of the reinforced material, a cradle-to-gate approach was chosen for the first analysis, considering tow cases involving lightweight alloys with the aim of comparison between aluminium alloys, as reference, and magnesium alloy. The consequence of the fuel savings caused by the lightweight material usage were carried out as a secondary analysis. The boundaries are depicted in Fig. 3.

2.3.3. *Life cycle inventory (LCI)*

The analysis for the LCI was divided into two levels. Firstly, a background analysis containing secondary standardised data, used in the models for all the life cycle steps including all upstream processes, to provide energy and material inputs for magnesium and aluminium production (described in the introduction section). Secondly, a foreground analysis, containing primary detailed data. The latter considers the relevant inputs and outputs associated with the die casting processes to produce the component reinforced with the submicrometre particles. All environmental models were performed using SimaPro v8.0 software.

2.3.3.1. *Background analysis: Metal production.* For this analysis, a consistent in-house database, comparable with commercial databases such as ecoinvent version 3, was used to provide the most relevant, reliable, transparent, and accessible LCI secondary data. The analysis started with the data shown in Fig. 4, which shows that the primary production of aluminium and magnesium is dominated by China. It can be seen that in 2012 China produced 75% and 34% of the global magnesium and aluminium in the world, respectively. Moreover, the production of primary magnesium (2012) is confined to only ten countries [35,36]. It seems that no European countries are currently listed among the main primary magnesium producing countries.

These data cannot be overlooked, since the analysis of the life cycle depends significantly on primary production allocation. In particular, this depends on the available energy sources in each country, which significantly influence the preparation of a complete database about the process for production of the aforementioned metals. Thus, the technology

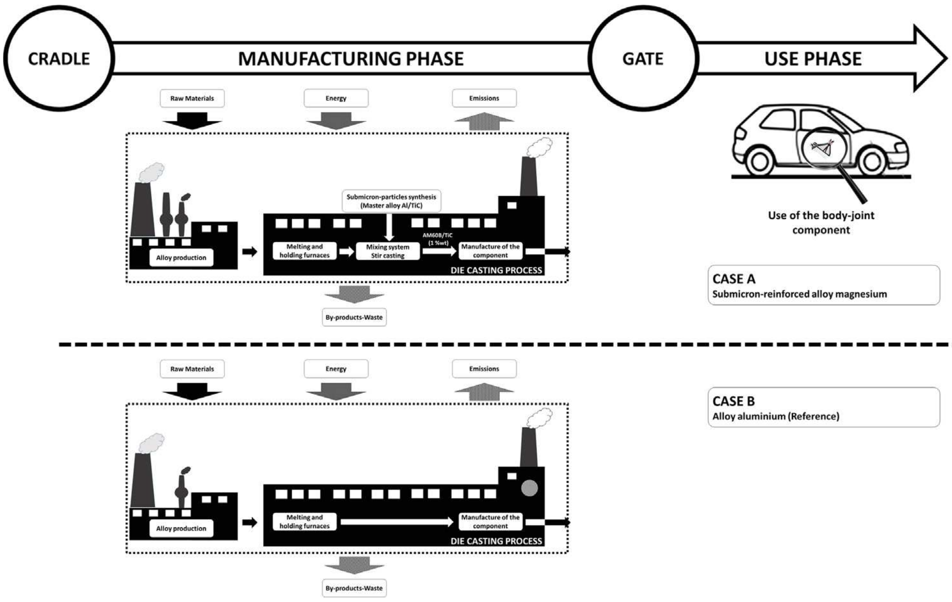


Fig. 3 – Description of the system boundaries in the study of life cycle for the passenger vehicle component fabricated using magnesium alloy reinforced with the submicrometre particles.

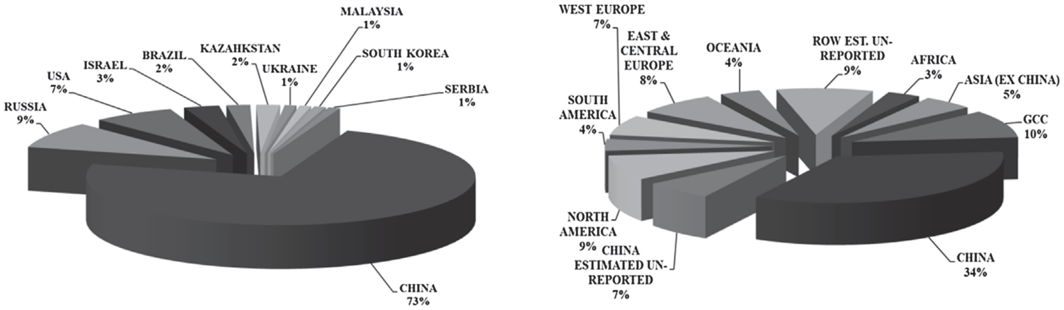


Fig. 4 – Production of magnesium (left) and aluminium (right), 2012 [26,27].

for environmental modelling assumed in this study is performed considering the standard aluminium technology in Europe for primary production.

Nevertheless, in case of magnesium, the main technologies across the globe should be considered. Therefore, two scenarios for the primary production of magnesium were taken into account in this study; the first consisting of the main process for producing magnesium in China, namely, the Pidgeon process, and the second a magnesium production plant based on electrolysis from resources like seawater and dolomite [37].

The authors have considered these two scenarios because of the marked differences between the two production technologies for primary magnesium. To aid understanding, a brief description of the production processes considered for these two metals (Al and Mg) and their alloying are described in the introduction section.

2.3.3.2. Foreground analysis: Die casting process to produce the Al and Mg alloy components. Die casting is the process analysed to produce the component either with aluminium or magnesium reinforced with the submicrometre particles. It is a manufacturing process normally featuring two furnaces, a melting and holding furnace. The solid metal is melted in the melting furnace, before being transferred to the holding furnace, in which the temperature is kept constant with a lower power to avoid its solidification. The molten metal is transferred from the holding furnace to a shot sleeve in order to be injected into the die cavity under a high pressure to obtain the desired casting [38]. The process includes machining and cooling stages (additional stages) after the solidification stage, in which the part is deburred in order to remove excess metal. The remaining metal can be melted again in future cycles.

In order to compare the magnesium die casting with the aluminium process, the materials and energy balance were calculated for the functional unit. In both cases, the weight of the component is the reference flow used in the energy and material flow model. The weights were 1.85 kg and 2.85 kg for the magnesium and aluminium components, respectively.

Moreover, it is important to highlight that the reinforced magnesium die casting process has an additional stage (see case A in Fig. 3), which is the system mixing. Therefore, this and the Al/TiC synthesis stage were taken into account for the LCI. Detailed information about the LCI is not shown here because of confidentiality issues, however the main magnesium LCI data referred to for the functional unit are summarised in Table 2.

Table 2 – Main inputs and outputs relevant for environmental analysis of the die casting magnesium.

Functional unit = 1 product final part = 1.85 kg		
Inputs		
Alloy magnesium (AM60)	2.1	kg/unit
Al/TiC master alloy	0.0325	kg/unit
Annual water	4.9×10^{-4}	m^3/unit
N_2	1.84×10^{-4}	m^3/unit
SO_2	3.60×10^{-6}	m^3/unit
Outputs		
N_2	1.8×10^{-4}	m^3/unit
SO_2	3.6×10^{-6}	m^3/unit
Produced dross	0.011	kg/unit
Dirty aluminium	0.0013	kg/unit
Final product	1.85	kg/unit
Water	4.9×10^{-4}	m^3/unit
Energy		
Melting and pre-heating furnace (Electricity)	1.1	kWh/unit
Holding furnace (Electricity)	0.8	kWh/unit
SHS process (Electricity)	0.2	kWh/unit
Mixing system (Electricity)	0.5	kWh/unit

2.3.4. *Savings calculation of CO_2 eq.*

A vehicle using parts from lightweight materials can have a reduced weight and therefore save fuel during vehicle operation. This results in a direct reduction in CO_2 eq. emissions during vehicle operation. Emissions of CO_2 and other GHGs decrease in proportion to the decrease in fuel consumption. It is worth noting that this does not apply to other emissions, such as particles or nitrogen oxides, because the development of emissions provoked by fuel savings is ambiguous.

Thus, the equation shown below, based on the work of Koffler and Rohde-Brandenburger [39], has been used for calculating the fuel saving in this study.

$$\text{Saved fuel} = (m - m_{ref}) \times R \times 0.01 \tag{1}$$

The saved fuel has units of fuel saving [l/100 km], m and m_{ref} are the masses of the alternative and reference components in kg, respectively. The constant R is the fuel reduction coefficient expressed in units [l/(100 km × 100 kg)]. The value of the fuel reduction coefficient is also based on the work of Koffler and Rohde-Brandenburger [39]. They reported a value of 0.35 l/100 km based on the standard new European driving cycle (NEDC) for passenger cars. This parameter is important, since it reflects the implications in terms of energy efficiency of the use of the magnesium component in an optimised vehicle. According to the aforementioned authors, the parameter R includes secondary measures when the energy consumption is calculated based on the NEDC.

2.3.5. *Cut-off criteria*

The following cut-off criteria were used in the environmental analysis to ensure that all relevant environmental impacts were represented in the study:

(i) Materials: flows of less than 1% of the cumulative mass of all the inputs and outputs of the LCI model, depending on the type of flow, were excluded because their environmental impact is negligible.
(ii) Energy: flows of less than 1% of the cumulative energy of all the inputs and outputs of the LCI model, depending on the type of flow, were excluded because their environmental relevance is negligible.

However, it was ensured that the sum of neglected material flows did not exceed 5% of the mass, energy, or environmental relevance. These criteria were established based on an in-depth analysis of the system using the operational energy and mass balances for the processes involved.

3. Results and discussion

3.1. *Technical evaluation*

The results obtained in the tensile tests confirm the benefits of adding the submicrometre TiC particulates to the magnesium alloy. The addition of 1 wt.% of particles leads to improvements of 11% and 18% in the yield stress and ultimate tensile strength values respectively and 30% in the ductility of the material. The microstructural analysis shows the presence of some small TiC aggregates but the balance of the effect of the presence TiC particulates is positive. TiC particulates appear both in the centre of the aluminium grains and the intergranular region and should contribute to strengthen the alloy by hindering the movement of dislocations through the Hall-Petch effect related to the decrease in the grain size, Orowan effect and the increase in the density of dislocations during the solidification stage due to the coefficient of thermal expansion (CTE) mismatch of the AM60 matrix and TiC.

3.1.1. *Material characterisation*

The production of the final reinforced AM60/(TiC 1 wt.%) material was carried out in two stages. Firstly, production of the Al/(TiC 60 wt.%) master alloy by SHS and subsequently production of the AM60/(TiC 1 wt.%) reinforced alloy by stir casting. Following the microstructures of the materials obtained in each stage are shown and discussed. Fig. 5 shows the physical aspect of the material obtained after the SHS process.

3.1.1.1. Characterisation of Al/TiC master compound. Fig. 6a shows the microstructure of the Al/TiC (30:70) compound obtained by SHS in the preliminary trials. TiC particulates appear as round particulates coated and surrounded by pure aluminium that is present in 30 wt.%. The microscopy observation of the samples shows that the self-propagating reaction has taken place correctly and there is neither any presence of loose Ti or C particulates nor any secondary product.

The size of the TiC particulates ranges from 0.5 up to 7 μm. The surface of TiC particulates is clean. Particulates are well wetted and appear covered by aluminium. Furthermore, there is not any clue of other secondary reactions that could lead to the formation of $TiAl_3$ or Al_4C_3 particulates. These observations are further confirmed with the results of the XRD analysis (see Fig. 7) where only Al and TiC appear. Nonetheless the goal of the work was to produce submicrometre-sized TiC particles and therefore the process to obtain a second generation of particulates was developed.

Fig. 5 – Al/TiC master alloy produced by SHS containing 40 wt.% of pure free aluminium manually crushed before the ball milling process.

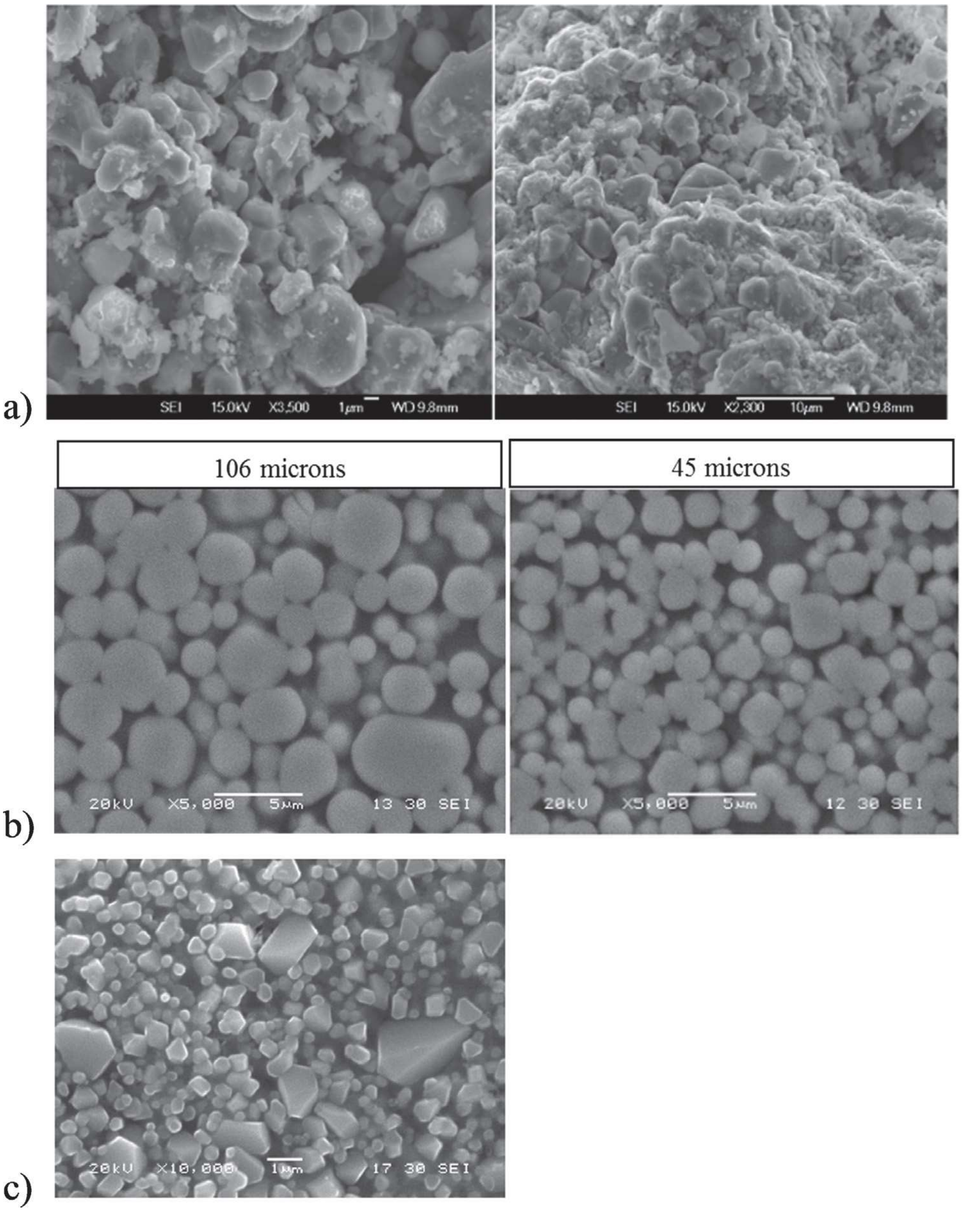


Fig. 6 – (a) Scanning electron micrograph of Al/TiC powders in a 30/70 ratio. The size of the TiC particulates ranges from 0.5 up to 7 μm, (b) Scanning electron micrograph of Al/TiC powder with Ti powder of 106 and 45 μm, (c) Scanning electron micrograph of TiC sample showing submicrometre-sized particles produced by SHS mixed with large particulates.

3.1.1.2. *Development of the second generation of submicrometre-sized TiC*. Two different approaches were tried to achieve submicrometre-sized TiC particles. On the one hand the use of elemental Ti powders having smaller sizes and on the other hand the acceleration of the solidification stage immediately after the end of the combustion in the SHS process so that the particles could not coalesce and grow within the reaction chamber.

Previous experiences accumulated by the authors had shown that the size of the initial powder of Ti used in the SHS reaction could have a direct influence on the size of the TiC powders obtained after the process. When using Ti powder with a size of 150 μm the average size of TiC particulates was around 1.5 μm (see Fig. 6a). The size decreased down to 0.5–5 μm when Ti powders of 106 μm were used (see Fig. 6b) and this size got further decreased with smaller Ti grain sizes 0.3–3 μm. Even so it was also observed that the difference of the TiC particulates obtained with Ti powders of 45 and 25 μm was minimal and therefore the size of 45 μm was selected for further tests.

Results of the second approach to decrease the cooling time of the reaction and consequently the size of TiC particulates are shown now. Different methods were tested to implement this concept, the application of compressed cold air and argon into the reaction chamber and increasing the aluminium content in the initial mixture of reactants.

The former showed to be difficult to implement and furthermore did not provide any apparent decrease in the TiC particulates. It was difficult to know the precise moment of

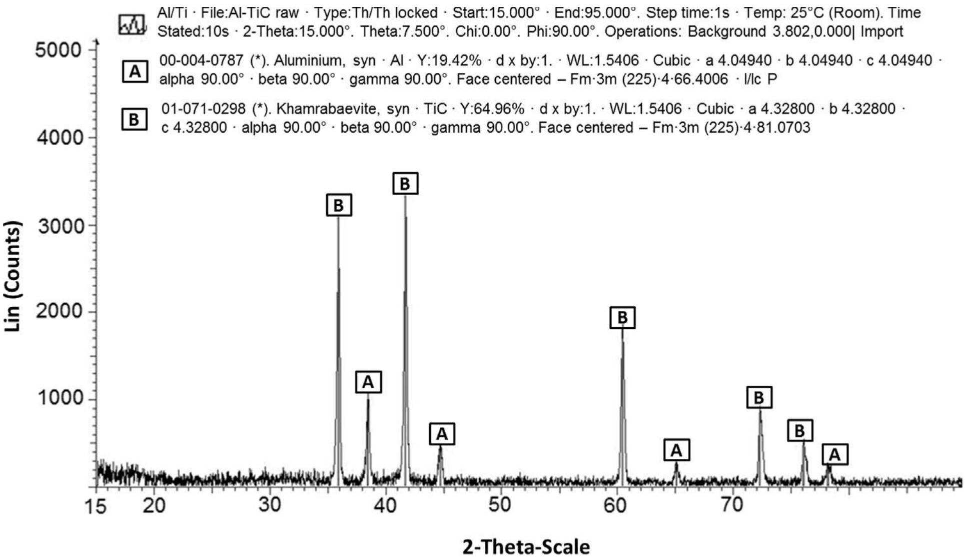


Fig. 7 – XRD pattern of Al/TiC powders.

the end of the reaction and therefore when to introduce the cold fluid into the chamber without disrupting the synthesis. The increase in the amount of aluminium in the initial mixture of Ti, C and Al powders resulted in a better approach. It was seen that the higher the aluminium content the smaller the size of the TiC particulates created. This is thought to be related to the increase in the heat dissipation capacity of the mixture as well as the decrease in the number of Ti and C atoms available that may diffuse into the TiC nuclei during the reaction. This option had a clear limit though. Above an amount of 40% of aluminium powders in the initial mixture, the reaction between Ti and C was no longer auto-propagated and the SHS synthesis did not proceed correctly. Eventually, the master alloy was prepared in the conditions that led to the lowest TiC particulates size, i.e. by using Ti powder of 45 μm and by incorporating 40% of aluminium into the initial mixture of Ti and black carbon powders. Fig. 6c shows a typical microstructure of the obtained powders. Small and large particulates with different shapes may be seen but most of the TiC particulates present a spherical shape and size lower than 500 nm.

As can be seen in Fig. 8, the study of the size distribution of the samples confirmed that most of the particulates were submicrometre-sized even though the presence of particulates larger than 1 μm may be appreciated. 47.3% of the particulates presented a size lower than 500 nm and only 22.4% of the particulates are bigger than 1 μm.

3.1.1.3. *Microstructure of the TiC reinforced magnesium alloy.*

Fig. 9 shows SEM images of the microstructure of the TiC reinforced magnesium alloy. It is appreciated that the border between the metal and the particulates is clean, there seems to be not any chemical reaction between both phases. Individual particulates can be seen both at the grain boundaries and within the grains. There are also small clusters that are located in the boundaries together with other typical secondary phases of the alloy. Stir casting process is known to help disperse ceramic particulates within the liquid metal but it may also create porosity and defects within the melt. The existence of agglomerations and increase of porosity is thought to be the main reason to explain the loss of properties appreciated when the incorporation of more than 1 wt.% particulates is tried. In fact, the improvement of properties shown by the samples containing 1 wt.% of particulates is really low in comparison with the sample with only 0.2 wt.%. The AM60 + TiC 0.2 wt.% samples present an increase of 5% in yield stress, 10% in UTS and up to 30% in terms of ductility when compared to the reference non-reinforced alloy: This increase is much limited when higher TiC contents are tried to be incorporated.

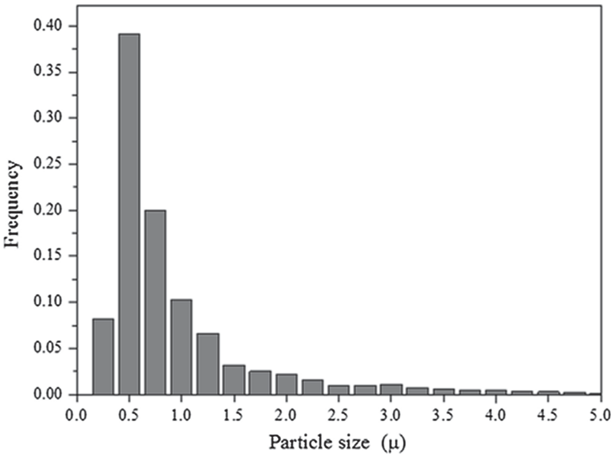


Fig. 8 – Size distribution of "second generation" TiC particulates obtained with Ti powder with an average size of 45 μm and 40% of aluminium in the initial mixture of Al/Ti/C.

3.1.2. *Mechanical properties*

Table 3 provides the results of the tensile tests of specimens containing AM60 alloy and different percentages of TiC particulates incorporated to the matrix through the stir casting process. Results of non-reinforced AM60 alloy obtained

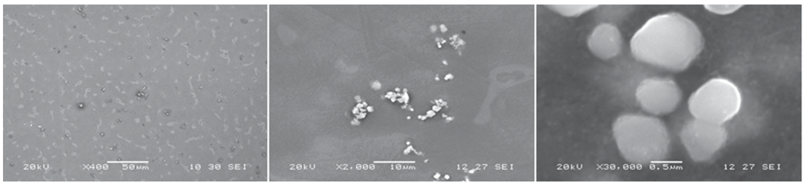


Fig. 9 – SEM images of the microstructure of the TiC reinforced magnesium alloy.

Table 3 – Mechanical properties of submicron reinforced AM60 alloy. Standard deviation values are provided in parenthesis.

Materials[a]	Yield stress (MPa)	Ultimate tensile strength (MPa)	Elongation (%)
AM60 (reference)	106.25 (20.81)	193.00 (34.26)	7.25 (3.23)
AM60 + TiC 0.2 wt.%	111.67 (4.95)	214.67 (24.26)	9.67 (2.83)
AM60 + TiC 1 wt.%	118.00 (10.50)	227.00 (6.00)	9.43 (0.46)
AM60 + TiC 1.5 wt.%	109.27 (17.76)	200.05 (26.34)	6.50 (2.87)

[a] All samples cast by gravity casting at 700 °C into a metallic mould.

with the same mould and casting parameters are provided for comparison purposes.

The analysis of the values points out that the addition of TiC particulates provides and improvement of properties as foreseen. Adding only 0.2% of particulates leads to an increase in around 5% in the mechanical strength and 30% in the ductility of the alloy. The mechanical strength further increases when the amount of particulates is increased up to 1 wt.% even though the ductility value presents a slight decrease and the samples containing 1.5 wt.% of TiC present worse results, even lower than those obtained with 0.2 wt.%. On the other hand, the analysis of the microstructures shows that the TiC particulates induce a decrease in the size of the magnesium grain. These results are in agreement with works based on submicrometre and ano reinforced magnesium alloys.

In contrast to composites with particulates larger than 1 μm, the ductility of the material seems to increase. The presence of TiC particulates leads to a decrease in the grain size and introduce stress fields due to the mismatch in CTE. They may also act as barriers to the movement of the dislocations through the phenomenon known as Orowan strengthening that explains that dislocations cannot advance due to the presence of well dispersed hard phases that must be overpassed and therefore act as barriers to their free movement. The presence of the dispersed particles may also induce the creation of cleavage cracks that may dissipate stress concentrations near the crack front.

These positive effects seem to fade when the content of TiC particulates overcomes 1 wt.%. This may be related to porosity of the samples due to the incorporation of oxides during the stir casting process of such composites.

3.2. *Environmental analysis*

3.2.1. *Evaluation of case study A: component manufacturing based on magnesium alloy*

Table 4 shows results concerning the environmental implications associated with the alloy production stage. On the one hand, it can be seen that in most of impact indicators, the process to produce magnesium (Pidgeon) has higher percentage when compared to the process carried out in the Norwegian plant. In particular, the climate change category shows a higher impact in the case of magnesium production by the Pidgeon process. This result can be associated with the source of energy required for the Pidgeon process. Fig. 10 shows the network for both process (Norwegian plant and Pidgeon process) containing the steps that contribute to the total CO_2 kg eq. In order to highlight the main product, the network's products are partially shown with a cut-off of node below 0.6%.

Thus, it can be observed that main contribution for both processes is focused on the electricity production where the main difference in terms of CO_2 eq. can be found in this point. This is corroborated when the climate change category of electricity production for both routes are compared and evaluated by the same method so far used (RECIPE). The CO_2 eq. emission per kWh of the Pidgeon process is 3 times higher than that emitted by the Norwegian plant, which leads to higher impact in terms of the climate change category.

The result above is consistent with the results of the study carried out by Simone et al. [33,40], which mention that the emissions from upstream in the Pidgeon process are attributed to the energy-intensive production. In particular, ferrosilicon (FeSi) is considered to be the largest contributor to overall emissions since a high amount energy (electricity) is used for its production. These authors also conclude that when the primary magnesium is obtained from electrolysis process, less GHG are emitted when compared to the Pidgeon process.

A similar analysis can be applied to the aluminium production process to explain why it reflects lower value of CO_2 eq. when compared to both processes to produce magnesium previously studied. As the magnesium produced by the Norwegian plant shows to have around 65% of indicators lower than aluminium production technology, the magnesium technology through the electrolysis method is the more suitable to obtain this light metal to be used in the component manufacturing.

Based on this premise, the magnesium through Norwegian plant has been considered for environmental modelling of the magnesium component manufacturing. Thus, and as

Table 4 – Results on environmental impact for the primary alloy production (aluminium and magnesium).

Impact category	Units	Aluminium production	Magnesium production (Norwegian plant)	Magnesium production (Pidgeon process)
Climate change	kg CO_2 eq	11.89	22.60	85.41
Ozone depletion	kg CFC-11 eq	7.14×10^{-7}	$3.13 \times 10^{-\circ 5}$	7.24×10^{-7}
Terrestrial acidification	kg SO_2eq	0.047	0.025	0.126
Freshwater eutrophication	kg P eq	0.005	0.0006	0.009
Marine eutrophication	kg N eq	0.002	0.006	0.015
Human toxicity	kg 1,4-DB eq	0.372	0.070	0.707
Photochemical oxidant formation	kg NMVOC	0.028	0.040	0.064
Particulate matter formation	kg PM10 eq	0.023	0.008	0.043
Terrestrial ecotoxicity	kg 1,4-DB eq	5.98×10^{-4}	$8.5 \times 10^{-\circ 5}$	4.31×10^{-4}
Freshwater ecotoxicity	kg 1,4-DB eq	0.002	0.0007	0.0030
Marine ecotoxicity	kg 1,4-DB eq	0.007	0.001	0.005
Ionising radiation	kBq U235 eq	3.040	0.412	0.745
Agricultural land occupation	m^2a	0.021	1.908	0.036
Urban land occupation	m^2a	0.025	0.022	0.165
Natural land transformation	m^2	-3.85×10^{-5}	1.00×10^{-4}	6.40×10^{-4}
Water depletion	m^3	321.13	15.94	21.46
Fossil depletion	kg oil eq	0.017	0.002	0.120

observed in Fig. 11a, the manufacturing process of non-reinforced magnesium component shows greater CO_2 eq. (climate change indicator) when compared to the aluminium component. This result is due to the magnesium alloy production with the higher energy consumption. In fact, when the environmental modelling was analysed for each stage, the alloy magnesium production phase resulted to have higher environmental burden than the die casting process for all indicators (disaggregated results are not shown here).

Nevertheless, the opposite occurs for most other impact indicators when comparing the magnesium component through Norwegian plant with aluminium. This corroborates that magnesium would be a good candidate among lightweight materials for the automotive industry.

3.2.2. *Evaluation of case study B: component manufacturing based on magnesium alloy reinforced with submicrometre-sized particles*

The results in previous section allow concluding that the process for magnesium alloy production has a significant influence in the life cycle of a vehicle component based on magnesium. Under this scenario, the environmental modelling described in this section also considers that the magnesium metal produced from electrolysis method to be used in the magnesium alloy, since, the process for manufacturing the component, using magnesium produced by Norks Hydro, would have a lower CO_2 eq. emissions compared to the Pidgeon process.

Thus, Fig. 11b depicts the environmental results of the three stages for vehicle component manufacturing using the magnesium alloy reinforced with submicrometre-sized particles: Al/(TiC 1 wt.%) synthesis stage, the mixing process and the component manufacturing. Firstly, it can be observed that the mixing step reveals higher percentages in all environmental categories. This is consistent with results shown in previous section, since, the mixing system stage considers the environmental burden due to the magnesium alloy production stage and, as explained above, this stage provokes a high environmental impact. By contrast, the synthesis of Al/(TiC 1 wt.%) shows lower percentages along the environmental indicators.

One may think that this is not the expected result, because of the use of submicrometre materials, such as submicrometre-sized titanium could have impacts in terms of toxicity. Although elemental titanium is of a low order of toxicity, laboratory animals (rats) exposed to titanium compounds via inhalation have developed small-localized areas of dark-coloured dust deposits in the lungs. Moreover, an excessive exposure in humans may lead to neuro-inflammation, further brain injury with a spatial recognition memory and loco-motor activity impairment [41]. Thus, it is important to highlight that the amount used in the magnesium alloy reinforced with submicrometre-sized particles is too small to determine the environmental implications through the methodology proposed in this study. In fact, in order to determine the potential environmental risks caused by the use of these materials, a further environmental modelling should be developed, in which, the environmental effects should be analysed by means of a particular LCA at submicrometre scale, taking into account the chemical and biological interactions of submicrometre-particles with their surroundings.

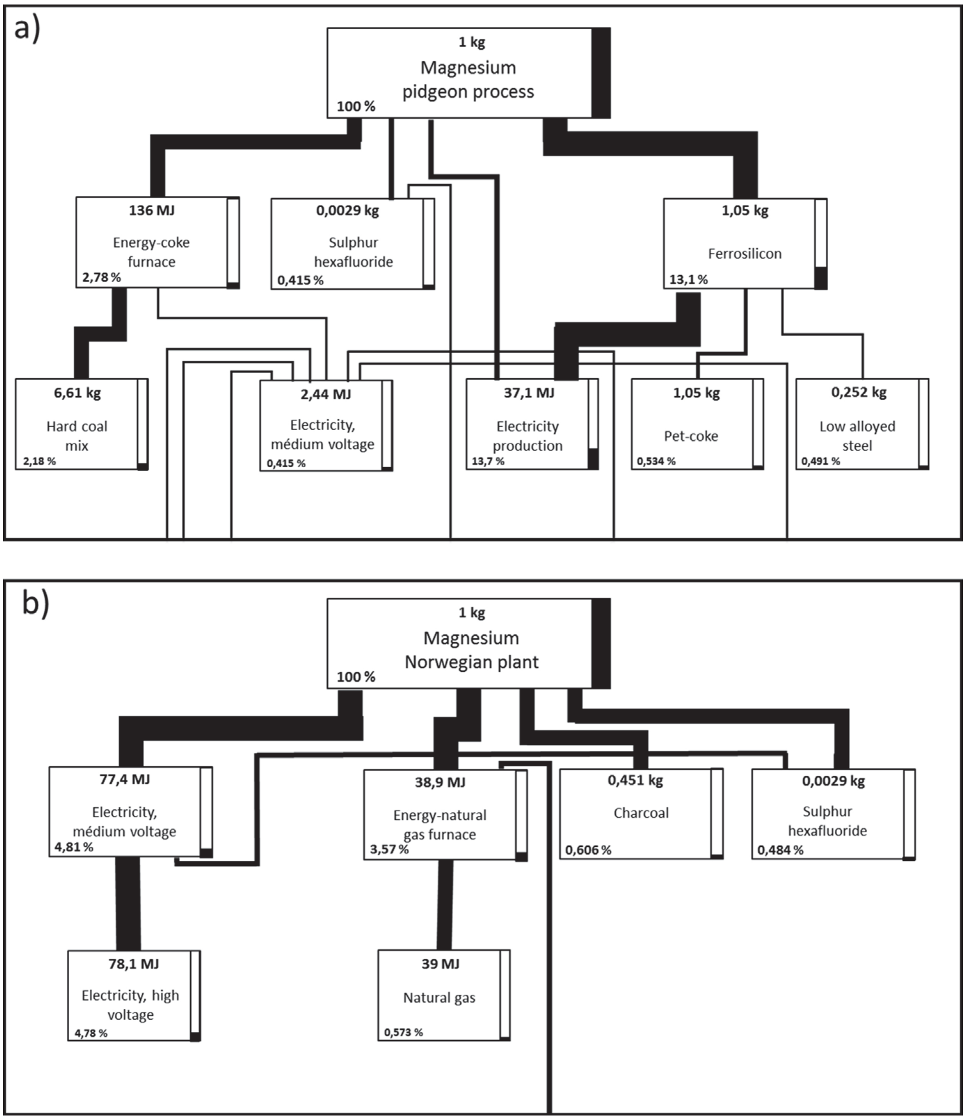


Fig. 10 – Network of products that contribute the total CO_2 eq of the magnesium production: (a) Pidgeon process (b) Norwegian plant. In order to highlight the main product, the network's products are partially shown with a cut-off of node below 0.6%.

In general, it can be said that, besides the positive effect of the Al/(TiC 1 wt.%) on mechanical properties, the incorporation of the submicrometre particles into matrix magnesium alloy has a low environmental impact when compared to other stages involved in the manufacturing process of the reinforced magnesium alloy component. However, major technological progresses are needed to develop a process to produce primary magnesium with minimum CO_2 emissions considering even a magnesium recycling process.

3.2.3. *Comparison of case A and B*

After the analysis of each case study, Fig. 12 shows the comparison of both cases, the environmental results for one component manufactured based on reinforced magnesium alloy (case A) and one component manufactured based on aluminium alloy (case B).

As it can be seen, it is not possible to conclude which case study is more environmentally friendly, since this conclusion would depend on the indicator analysed. Nevertheless, it can be summarised that except five environmental indicators (climate change, marine eutrophication, ionising radiation, agricultural land occupation and natural transformation), the rest reveal that the component manufactured using the aluminium alloy has a higher impact when compared to magnesium. The difference is particularly remarkable in case of freshwater eutrophication, human toxicity, terrestrial and marine ecotoxicity, where the impact associated with the reinforced magnesium component is approximately lower 20% than the impact of the aluminium component.

Nevertheless, one of the main indicators that are considered in the road transport is the climate change. In this study, the reinforced magnesium alloy has a higher impact compared to the component manufacturing using aluminium. For that reason, it has been necessary to consider including the study of the use phase, in which, the fuel saving caused by

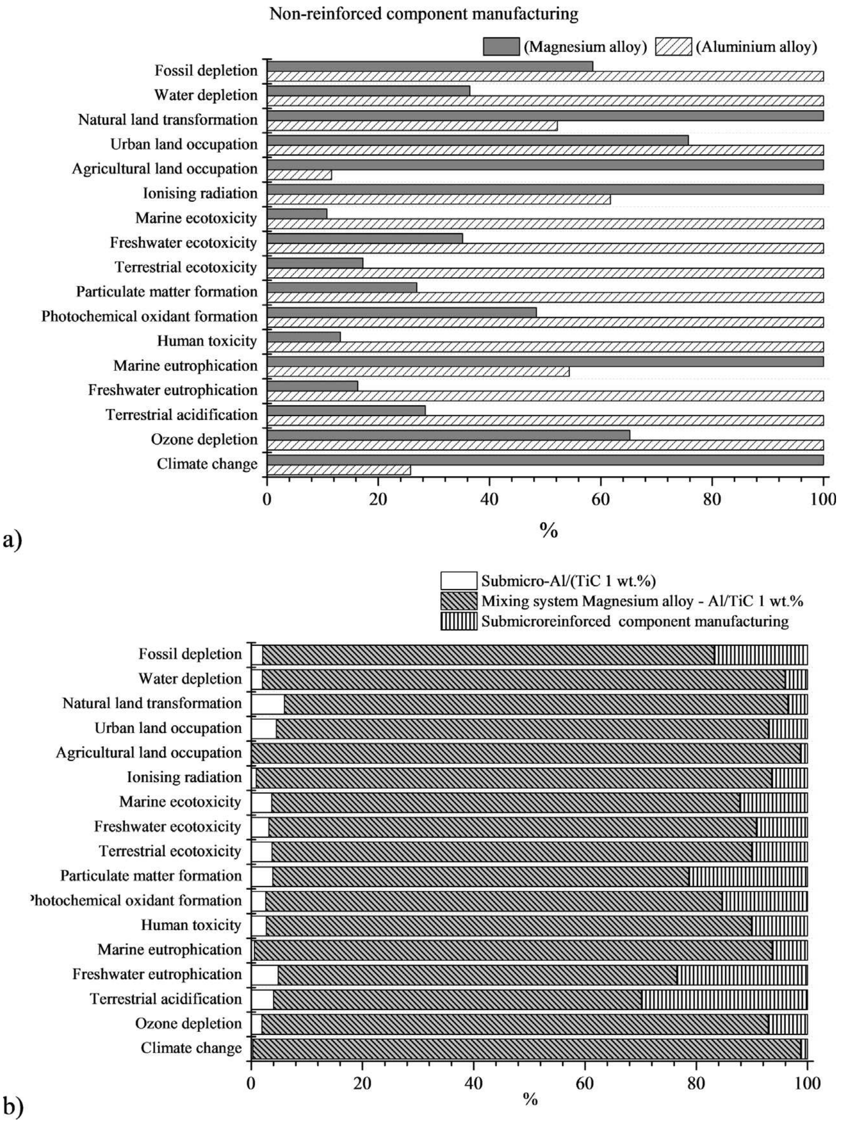


Fig. 11 – (a) Results on environmental impact categories for non-reinforced component manufacturing, (b) Environmental burdens associated with the different stages of the component manufacturing based on magnesium alloy reinforced with submicrometre-sized particles.

the lightweight component, can reduce the CO_2 eq. emissions and therefore determine if the higher environmental impact associated with the magnesium component manufacturing is offset.

3.2.4. *Use phase scenario analysis*

As observed in Table 4, the results show that magnesium production technologies lead higher CO_2 eq. when compared to the aluminium production, which is mainly attributed to the high energy consumption generating considerable GHG emissions. Therefore, the special interest and contribution of this work is focused in increasing and broadening the applications of magnesium (as a lightweight material) with improved mechanical properties in the automotive sector.

It is known that the amount of fuel and emission savings during the use phase depends largely on the weight reduction resulting from the material substitution. Thereby, from this basis of calculation form based on the fuel consumption savings, the above statement can be evidenced in Fig. 13. There the difference between CO_2 eq. emissions respect to the reference component is depicted versus the driven millage of a car with an assumed lifetime of 200,000 km (the reference case shown in the figure is represented as the aluminium baseline (dotted line)). A point around 28,000 km can be observed at which the CO_2 eq. emissions are compensated with respect to the component manufacturing including the production of metal alloy. This point can be considered as a break-even point.

From this point, the use of a lightweight component led to negative values of Δ kg CO_2 eq., which indicates benefits from the environmental point of view. This is an important result indicating that the use of reinforced magnesium in

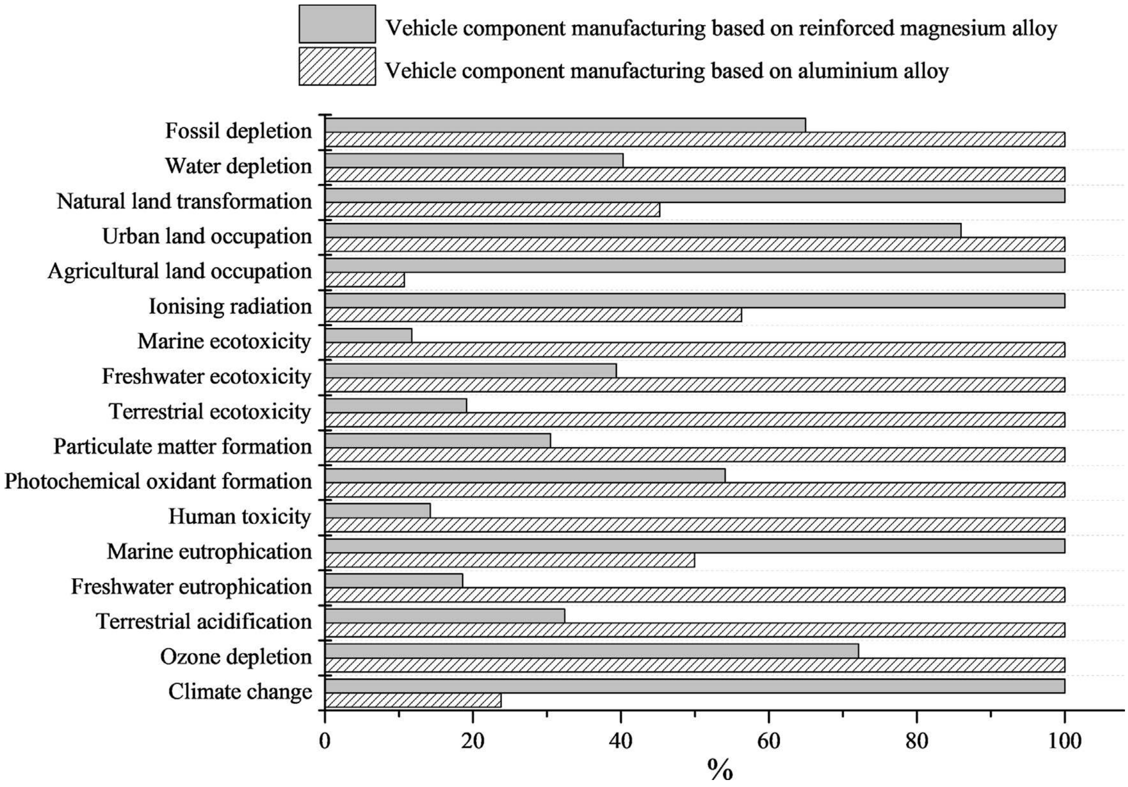


Fig. 12 – Comparison of the environmental results obtained for case A (one component manufactured based on reinforced magnesium alloy) and case B (one component manufactured based on aluminium alloy).

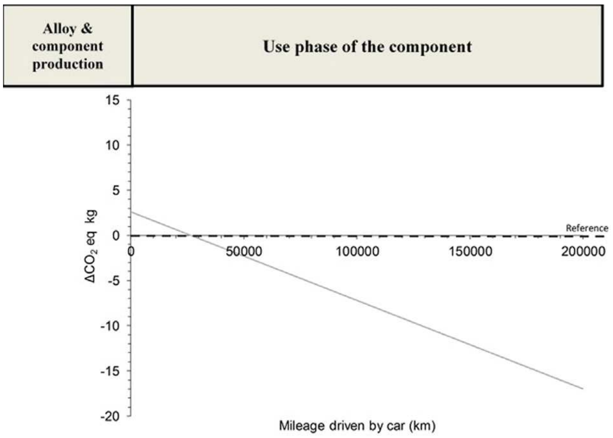


Fig. 13 – CO_2 eq. savings in the use phase of the generic body joint component respect to the aluminium component (Reference).

automotive components not only improves the mechanical properties, but it mitigates the environmental burden in terms of climate change.

The above results are comparable with the Simone et al.'s study [33]. They raise several scenarios for primary magnesium production based on similar technologies, where their high environmental burdens are compensated during the use stage of the vehicle component manufactured with magnesium alloy.

Nevertheless, it is worth noting that obtaining a break-even point at low mileage driven depends considerably of initial conditions. In other words, the CO_2 eq. impact attributed to the component manufacturing (intersection point on the axis of ordinates) will be offset in a reasonable mileage depending on the weight reduction in the vehicle by using a magnesium component as well as of the novel technology for its manufacture. Thus, the environmental burden in its life cycle (from cradle-to-gate including the use phase) will be balanced.

4. Conclusions

This study analysed simultaneously the technical and environmental performance of a novel developed magnesium alloy reinforced with submicrometre-sized TiC particles to produce automotive components. The AM60/(TiC 1 wt.%) master compound was produced through self-propagating high-temperature synthesis (SHS) process and embedded in magnesium alloy by a mechanical method. In general, the study showed positive results in both perspectives, the technical and environmental evaluations.

On the one hand, the technical evaluation analysed the most representative physical and mechanical properties. In case of physical properties, the results confirmed that during the SHS self-propagation reaction no presence of any loose of Ti or C particles or any other secondary components was found. The submicrometre-sized TiC production, a good size distribution of the particles (as was shown by SEM and XRD analysis), was achieved. In depth, most of them were submicrometre-sized lower than 500 nanometres (more than 47% of the particles) and spherical shaped. Only 22.4% of the particles obtained were bigger than $1\,\mu m$. In addition, results also confirmed the good performance of the material since it was shown that the addition of up to 1 wt.% of TiC increased 11% the yield stress, 18% the ultimate tensile strength and 30% the ductility of the material with regards to the case of initial magnesium alloy.

On the other hand, the environmental evaluation revealed that the incorporation of this submicrometre-particle into a matrix magnesium alloy had a low environmental impact when compared to other stages involved in the manufacturing process of the component, in particular, the magnesium production. This latter showed highest environmental impact when compared to the aluminium process, more than 70% of the indicators in the Pidgeon method.

Nevertheless, comparing the final automotive component, made of reinforced magnesium through the electrolysis method using the alternative R134 gas cover, with the aluminium component, the results revealed that the environmental impact associated with the reinforced magnesium component showed lower environmental (70% of the indicators analysed).

However, this was not observed in the case of the climate change indicator, where the production of the reinforced magnesium component entailed higher amount of CO_2 eq. than in the case of the aluminium component (around 90%). Therefore, the study included the use phase of the component where the lower weight of this component results in fuel saving in the car and, consequently, an associated reduction in the CO_2 eq., which offsets the environmental impact in terms of climate change induced in the magnesium production stage.

Conflicts of interest

The authors declare no conflicts of interest.

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