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(54) **Title:** A PROCESS FOR PRODUCING REINFORCED ALUMINUM-METAL MATRIX COMPOSITES

(57) **Abstract:** The present disclosure relates to a process for producing metallic carbide particulate reinforced aluminum alloy matrix composite. The present disclosure envisages generating in-situ carbon for reacting with the metal containing compound in the molten aluminum matrix to obtain metallic carbide particulate. A hydrocarbon compound is selected as a carbon source. The process of the present disclosure allows up to 20 % of the metallic carbide particulate reinforcement to be introduced in the aluminum matrix.



A PROCESS FOR PRODUCING REINFORCED ALUMINUM-METAL MATRIX COMPOSITES

FIELD OF DISCLOSURE

The present disclosure relates to a process for producing an aluminum-metal matrix composite.

The present disclosure particularly relates to a process for producing Titanium-Carbide (TiC) reinforced aluminum-metal matrix composite.

BACKGROUND

A metal matrix composite (MMC) is a composite material with at least two constituent parts, one being a metal. The other material may be a different metal or another material, such as a ceramic or organic compound. These composites are generally tailor-made depending upon the application requirements. The composite includes a reinforced material embedded in the metal matrix. The reinforced material can be synthesized externally and then embedded in the metal matrix or can be prepared in-situ in the metal matrix. Aluminum is a most preferred matrix for metal matrix composites due to its low density and capacity to be strengthened. One particular class of aluminum-based MMC that has gained popularity in the recent times is the titanium-carbide (TiC) particulate reinforced aluminum-metal matrix composite, especially wherein the TiC particulates are in-situ formed in the aluminum-metal matrix.

US 4,772,452, discloses a process for TiC reinforced aluminum matrix composites wherein the aluminum metal, titanium bearing compound and the carbide, all provided in the powder form are pre-mixed, compacted and further heated at a reaction temperature approximating melting point of the aluminum to produce the composite.

US 6,843,865 discloses a process for TiC reinforced aluminum matrix composites wherein the mixture of aluminum and titanium metals in its molten form is reacted with a halide of carbon to produce the composite. The reaction is carried out under vigorous mechanical stirring.

US 4,748,001 discloses a process for TiC reinforced aluminum matrix composites wherein the carbon powder preheated to 700⁰C is added to the molten mixture of aluminum and titanium metals and the melt is stirred vigorously at high temperature and additional processing is carried out at a very high temperature (1100 to 1400⁰C) to produce the desired composite. The melt is agitated by mechanical stirring.

Indian Patent Application No. 168/MUM/2010 discloses a method using pneumatic injection of a mixture of titanium bearing compounds and carbon containing material for the in-situ synthesis of TiC-aluminum composites. During synthesis of aluminum-titanium carbide matrix, graphite powder is used to react with titanium to form TiC.

US 4,808,372 discloses a process for producing a composite by introducing a gas into a molten composition comprising a matrix liquid and a refractory material-forming component and subsequently adding a reactive component

which reacts with the refractory material-forming component to cause the refractory material to disperse in the matrix liquid. The gas may be an inert carrier gas comprising a carbonaceous material such as methane which is introduced through a tube into the molten composition.

The composites so obtained have enhanced mechanical properties compared to aluminum metal and aluminum metal alloys and these composites find wide applications in transportation, electronics, and recreational products. However, one severe drawback with the known techniques is that it is very difficult to obtain a homogenous dispersion of the TiC particles in the aluminum metal matrix. This leads to a variation in composite properties not only from batch to batch but even within the sample. Also, the process is carried out at very high temperatures, typically in the range of 1100 – 1400 °C, for duration of 1 – 2 hours. This leads to high processing costs. Another drawback of the known processes is that they require preheating of the precursors to allow wetting of the powders in the melt. The powder size is also to be controlled within tight specifications to enable good mixing and wetting. Also, the processes can be used to obtain only up to 5 % particulate reinforcement, beyond which mixing is poor.

Therefore, there is felt a need to develop a composite having a higher amount of particulate reinforcement and uniform homogenous distribution of the particulate reinforcement for superior mechanical properties.

OBJECTS

Some of the objects of the present disclosure, which at least one embodiment herein satisfies, are as follows:

A main object of the present disclosure is to provide a process for producing metal carbide reinforced aluminum-metal matrix composites, which reduces the formation of carbon agglomerates in the composites.

Another object of the present disclosure is to provide a process for producing metal matrix composites which provides a fine and homogenous distribution of the carbide particulate in the aluminum-metal matrix.

Another object of the present disclosure is to provide a simple and cost-effective process for producing metal matrix composites.

Still another object of the present invention is to provide a process for producing metal matrix composites which requires low operating temperature and reduced processing times.

One more object of the present invention is to provide a process for producing metal matrix composites with high amount of particulate reinforcement.

Other objects and advantages of the present invention will be more apparent from the following description when read in conjunction with the accompanying figures, which are not intended to limit the scope of the present disclosure.

SUMMARY

In accordance with the present disclosure there is provided a process for producing a reinforced aluminum-metal matrix composite, said process comprising the following steps:

- a) providing a molten aluminum matrix maintained at a temperature in the range of 750⁰C to 1200⁰C;
- b) pneumatically injecting a metal containing compound into said molten matrix to form an alloy melt;
- c) injecting a hydrocarbon compound into said melt, to generate in-situ carbon for reacting with said melt, to obtain molten alloy containing metallic carbide particulate; and
- d) casting and solidifying said molten alloy.

Typically, said metal compound is injected into said molten matrix through a feeder attached to a submersible lance, said lance being immersed in said molten matrix.

Typically, the hydrocarbon compound is injected into said melt through a feeder attached to a submersible lance, said lance being immersed in said molten matrix.

Typically, the hydrocarbon compound is selected from the group consisting of paraffin and acetylene gas.

In one of the embodiment of the present disclosure, said metal compound is injected along with the hydrocarbon into said molten matrix.

Typically, said metal compound and the hydrocarbon compound is injected pneumatically using pressurized carrier gas.

Typically, the carrier gas is selected from the group consisting of argon and nitrogen.

In a preferred embodiment of the present disclosure, said metal compound is in the powder form.

Typically, said metal compound is a titanium compound.

Typically, the titanium compound is selected from the group consisting of potassium titanium fluoride and titanium oxide.

Typically, said molten matrix is maintained at a temperature in the range of 850⁰C to 1000⁰C.

Typically, the molten alloy formed in step c) is further agitated with the carrier gas for the uniform distribution of metallic carbide particulate in the molten alloy.

In one of the embodiment of the present disclosure, said molten matrix further comprises at least one alloying element selected from the group consisting of copper, zinc, magnesium and silicon.

In one of the embodiment of the present disclosure, the process further comprises the step of adding at least one alloying element to the molten alloy

containing metallic carbide particulate, said alloying element selected from the group consisting of copper, zinc, magnesium and silicon.

Another aspect of the present disclosure provides a reinforced aluminum-metal matrix composite having number of defects less than 15/100micron².

BRIEF DESCRIPTION OF THE ACCOMPANYING DRAWINGS:

Figure 1 illustrates XRD patterns of Aluminum-Titanium carbide (AL-TiC) composites prepared by using different carbon sources.

Figure 2 illustrates effect of alternate carbon sources like Graphite Powder and Paraffin Wax on tensile properties of Aluminum Matrix Composites.

DETAILED DESCRIPTION

The embodiments herein and the various features and advantageous details thereof are explained with reference to the non-limiting embodiments in the following description. Descriptions of well-known components and processing techniques are omitted so as to not unnecessarily obscure the embodiments herein. The examples used herein are intended merely to facilitate an understanding of ways in which the embodiments herein may be practiced and to further enable those of skill in the art to practice the embodiments herein. Accordingly, the examples should not be construed as limiting the scope of the embodiments herein.

The description herein after, of the specific embodiments will so fully reveal the general nature of the embodiments herein that others can, by applying current knowledge, readily modify and/or adapt for various applications such specific embodiments without departing from the generic concept, and, therefore, such adaptations and modifications should and are intended to be comprehended within the meaning and range of equivalents of the disclosed embodiments. It is to be understood that the phraseology or terminology employed herein is for the purpose of description and not of limitation. Therefore, while the embodiments herein have been described in terms of preferred embodiments, those skilled in the art will recognize that the embodiments herein can be practiced with modification within the spirit and scope of the embodiments as described herein.

Metal matrix composites (MMC) are tailor made material consisting a reinforcing material dispersed in a metal matrix. The matrix is a monolithic material into which the reinforcement is embedded. The reinforcement is provided to improve physical properties such as wear resistance, friction coefficient, or thermal conductivity of the metal.

Aluminum Matrix Composites (AMC) are used for manufacturing automotive parts (pistons, pushrods, brake components), brake rotors for high speed trains, bicycles, golf clubs, electronic substrates, cars for high voltage electrical cables.

Various methods are used to prepare MMC such as i) Solid state method, where powdered metal and reinforcement material are mixed and then bonded through a process of compaction, degassing, and thermo-mechanical treatment. ii) Liquid state method wherein reinforcement material is stirred into the molten

metal and allowed to solidify. iii) A chemical reaction between the reactants to form reinforcement material in-situ in metal matrix. iv) Vapor deposition wherein the fiber is passed through a thick cloud of vaporized metal, coating it.

Aluminum Matrix Composites are manufactured by the fabrication methods such as Powder metallurgy (sintering), Stir casting and Infiltration. Usually the reinforcement of Aluminum Matrix Composites results in high strength, high stiffness (modulus of elasticity), Low density, High thermal conductivity and excellent abrasion resistance of the reinforced metal compared to properties of pure metal.

The present disclosure is accomplished taking into account the above described goals and objects of the present disclosure.

Accordingly, the present disclosure envisages generating in-situ carbon for reacting with the metal containing compound. A hydrocarbon compound is selected as a carbon source.

The present disclosure provides a process for producing a reinforced aluminum-metal matrix composite which involves following steps:

- a) providing molten aluminum matrix maintained at a temperature in the range of 750⁰C to 1200⁰C;
- b) pneumatically injecting a metal containing compound into said molten matrix to form an alloy melt;
- c) injecting a hydrocarbon compound into said melt , to generate in-situ carbon for reacting with said melt , to obtain molten alloy containing metallic carbide particulate; and

d) casting and solidifying said molten alloy.

A pressurized injection lance is used to inject the metal bearing compound and the hydrocarbon compound in to the molten matrix. The hydrocarbon compound and the metal bearing compound may be injected simultaneously or sequentially or as a mixture into the molten matrix. An inert gas, preferably nitrogen or argon serves as a carrier gas. The lance is submerged in the bottom of the reactor system containing the molten aluminum.

In a preferred embodiment of the present disclosure, the hydrocarbon compound in the form of a liquid or gas. The hydrocarbon fluid undergoes spontaneous combustion on exposure to high temperature, thereby forming in-situ carbon which then reacts with the metal bearing compound in the molten aluminum matrix.

In a preferred embodiment of the present disclosure, the metal containing compound is in a powder form. The metal compound is a titanium compound selected from the group consisting of potassium titanium fluoride and titanium oxide.

The pressurized gas helps to agitate the melt to ensure intimate mixing, which enhances the reaction kinetics and lowers the processing temperature (800-1000°C) and processing time (5 to 60 min). The process thus avoids mechanical stirring which may lead to irregular particulate size. Improvement is also observed in the homogeneity of mechanical properties, e.g. hardness variation is < 5% within the casting. This process allows higher amount of reinforcement to be introduced in the melt (up to 20%), without compromising casting

integrity. Composites prepared by this process have a finer and more uniform distribution compared to those prepared by conventional route of mechanical stirring. Therefore for the same volume fraction of particles, composites according to the process described herein have superior mechanical properties. Also, use of the hydrocarbon compound instead of the carbon (graphite) powder reduces the occurrence of carbon agglomerates in the composites and reduces lance choking due to the accumulation of the graphite particles inside the lance. Since the flow rate and duration of hydrocarbon compound can be controlled independently of the titanium bearing compound feed stream, the process of the present disclosure allows more flexibility in controlling the different process parameters to improve composite quality.

In one embodiment of the present disclosure, the process is carried out using liquid paraffin as a source for generating carbon. Liquid paraffin is taken into a distributor column which is connected on one end to an argon cylinder and to an alumina lance on the other. The column is provided with a valve to control the flow rate of the liquid paraffin.

In another embodiment of the present disclosure, the process is carried out using acetylene gas as a source for generating carbon. Instead of a distribution column for addition of paraffin, a simple header is used for mixing the argon and acetylene gas stream. Individual valves provided in both the gas streams allow the argon/acetylene ration to be easily controlled.

In accordance with one of the embodiments of the present disclosure, the molten aluminum matrix may include alloying elements selected from the group consisting of silicon, zinc, magnesium or copper.

In accordance with one of the embodiments of the present disclosure, at least one of the alloying elements selected from the group consisting of silicon, zinc, magnesium or copper may be added during the process to further enhance the properties of reinforced aluminum matrix.

Depending on the process parameters, the carbon either forms metallic-carbide or leads to surface modification of Al_3M from long needles and plates to a more equi-axed structure.

The disclosure will now be described with respect to the following examples which do not limit the subject matter of the present disclosure in any way and only exemplifies the invention.

Example 1: Using Carbon powder

462 gm of aluminum metal was melted in a graphite crucible at 900°C. A mixture of Potassium titanium fluoride and carbon powder (97.3g of K_2TiF_6 and 7.5 g of C) was added to the molten aluminum using a screw feeder attached to an alumina lance immersed in the molten melt using argon as the carrier gas. After feeding the additives for 8 minutes, the screw feeder was switched off and the melt was mixed with argon stirring for additional 5 minutes. The amount of addition corresponds to a nominal addition of 10% TiC by volume. At the end of stirring, the crucible was removed from the furnace, the dross was skimmed off from the melt. The composite sample was cast in cast iron moulds.

Example 2: Using Acetylene Feedstock

18 kg of aluminum was melted in an induction furnace at 900°C. 10 kg of potassium titanium fluoride was added to the molted metal through a metallurgical lance using argon as a carrier gas. Acetylene gas was mixed with the argon gas and fed into the molten metal bath. The powder and gas mixture was injected in the melt for 30 minutes. The amount of additions corresponds to 20% TiC. At the end of the addition, the molten bath was allowed to react and cool to separate the metal from the dross. After removal of dross, predetermined quantity of silicon and magnesium master alloy were added to the melt to prepare Al-0.6% Si-0.6% Mg-20% TiC composites. The composite was then poured and cast in steel molds to produce extrusion billets and rolling ingots.

The composite was extruded at 400°C. Instead of a distribution column for addition of paraffin, a simple header is used for mixing the argon and acetylene gas stream. Individual valves in both the gas streams allow the argon/acetylene ratio to be easily controlled.

Tensile properties like yield strength (YS), ultimate tensile strength (UTS) and ductility (E%) of cast and extruded composite is as shown in Table 1.

Table 1:

Sl. No.	Sample description	As-cast			Extruded		
		UTS (Mpa)	YS (Mpa)	E%	UTS (Mpa)	YS (Mpa)	E%
263	0.6%Si-0.6%Mg/15%TiC/Acetylene	107	106	1	293	199	9

Example 3: Using Paraffin Feedstock

18 kg of aluminum was melted in an induction furnace at 900°C. 10 kg of potassium titanium fluoride was added to the molted metal through a metallurgical lance using argon as a carrier gas. Paraffin liquid or vapourised paraffin wax corresponding to 200% of the stoichiometric amount of carbon required was measured and poured into a distributor column. The column was connected on one end to an argon cylinder and to an alumina lance on the other. The column had a valve to control the flow rate of the paraffin wax. After feeding the additives for 8 minutes, the screw feeder was switched off and the melt was mixed with argon stirring for additional 5 minutes. The amount of addition corresponds to a nominal addition of 20% TiC by volume. At the end of stirring, the crucible was removed from the furnace, the dross was skimmed off from the melt. The composite sample was cast in cast iron moulds.

Figure 1 illustrates the formation of TiC using graphite and acetylene variants.

Figure 2 illustrates effect of paraffin and graphite powder on the tensile properties of the Aluminum matrix composites. Using Paraffin, marked improvement in ductility is observed over the graphite source. Furthermore it is seen that the standard deviations in strength is lower for inventive process

compared to prior art. The minor decrease in strength for the inventive process compared to prior art is attributed to lower reinforcement content.

XRD- pattern of Al-TiC composites prepared by using different carbon sources is depicted in Table 2

Table 2

	Angle	Intensity	Angle	Intensity	Angle	Intensity	Angle	Intensity	Angle	Intensity
Carbon powder (Graphite)	36.036	52.8	38.610	2565	39.377	473	42.107	143	44.863	1022
Acetylene	35.971	26.7	38.513	2767	39.247	521	42.034	140	44.764	1121
Paraffin	36.036	0	38.509	4753	39.200	942	41.992	184	44.765	2008

Table 3 illustrates the number of defects in Al-TiC composite prepared by using different carbon sources.

The defect density was measured by taking the cross section of the composite samples at three locations: top, middle and bottom of the cast ingot respectively. The cross section was then ground and polished for microscopic examination. The defect density was estimated by counting the number of defects in an area of 100 microns by 100 microns. The average defect size was estimated by measuring the average linear intercept size of the defects. Number of defects in Al-TiC composite prepared by graphite route was much higher compared to that prepared by acetylene route as reflected in Table 3. The size of defects in graphite route samples are also found slightly larger compared to acetylene route samples.

Table 3

Sample Id	Parameter	No. of defects at location			Average	Defect size (mm)	
		1st	2nd	3rd		Min	Max
1	Graphite	25	23	22	23	60	300
2	Acetylene	12	8	5	8	40	300

A reinforced aluminum-metal matrix composite prepared in accordance with the present disclosure is found to have number of defects less than 15/100micron².

As discussed earlier, carbon source has an effect on the defect density of the composite. This, in turn, affects the tensile properties like yield strength (ys), ultimate tensile strength (uts) and ductility (% elongation) of the composite. Effect of carbon sources like graphite powder and paraffin wax on tensile properties of aluminum matrix composites is shown in Table 4.

Table 4: Effect of carbon sources like graphite powder and paraffin wax on tensile properties of aluminum matrix composites

Table 4

	Ultimate Tensile Strength (UTS, MPa)	Yield Strength (YS, MPa)	Modulus (GPa)	% Elongation
Paraffin	107.3 ± 5.3	44.8 ± 5.5	77.2 ± 3.9	14.2 ± 7.4
Graphite	118.5 ± 10.6	57.5 ± 6.4	74.0 ± 4.2	8.0 ± 0.8

TECHNICAL ADVANTAGES

A process for producing metal matrix composites, particularly titanium-carbide (TiC) reinforced aluminum-metal matrix composite, as described in the present disclosure has several technical advantages including but not limited to the realization of:

- the process of the present disclosure helps in achieving homogenous mixing, thus, giving a uniform distribution of the particulate reinforcement in the metal matrix and thereby giving homogeneity in the mechanical properties of the composite, e.g. variation in the hardness of the composite is less than 5 %, within the casting;
- the reaction kinetics are enhanced due to the pneumatic injection which results in lowering the process temperature to 850 – 1000 °C and the process time to 5 – 20 minutes;
- the process of the present disclosure allows up to 20 % of the particulate reinforcement (TiC) to be introduced in aluminum matrix; and
- use of hydrocarbon fluid helps in reducing the formation of carbon agglomerates in the composite and also reduces lance choking due to accumulation of the carbon particles.

The numerical values mentioned for the various physical parameters, dimensions or quantities are only approximations and it is envisaged that the values higher/lower than the numerical values assigned to the parameters, dimensions or quantities fall within the scope of the invention, unless there is a statement in the specification specific to the contrary. Wherever a range of values is specified, a value up to 10% below and above the lowest and highest

numerical value respectively, of the specified range, is included in the scope of the invention.

In view of the wide variety of embodiments to which the principles of the present invention can be applied, it should be understood that the illustrated embodiments are exemplary only. While considerable emphasis has been placed herein on the particular features of this invention, it will be appreciated that various modifications can be made, and that many changes can be made in the preferred embodiments without departing from the principle of the invention. These and other modifications in the nature of the invention or the preferred embodiments will be apparent to those skilled in the art from the disclosure herein, whereby it is to be distinctly understood that the foregoing descriptive matter is to be interpreted merely as illustrative of the invention and not as a limitation.

Throughout this specification the word “comprise”, or variations such as “comprises” or “comprising”, will be understood to imply the inclusion of a stated element, integer or step, or group of elements, integers or steps, but not the exclusion of any other element, integer or step, or group of elements, integers or steps.

The use of the expression “at least” or “at least one” suggests the use of one or more elements or ingredients or quantities, as the use may be in the embodiment of the invention to achieve one or more of the desired objects or results.

Any discussion of documents, acts, materials, devices, articles or the like that has been included in this specification is solely for the purpose of providing a context for the invention. It is not to be taken as an admission that any or all of these matters form part of the prior art base or were common general knowledge in the field relevant to the invention as it existed anywhere before the priority date of this application.

While considerable emphasis has been placed herein on the specific steps of the preferred process, it will be appreciated that many steps can be made and that many changes can be made in the preferred steps without departing from the principles of the disclosure. These and other changes in the preferred steps of the disclosure will be apparent to those skilled in the art from the disclosure herein, whereby it is to be distinctly understood that the foregoing descriptive matter is to be interpreted merely as illustrative of the disclosure and not as a limitation.

Claims:

1. A process for producing reinforced aluminum-metal matrix composite, said process comprising the following steps:
 - a) providing a molten aluminum matrix maintained at a temperature in the range of 750⁰C to 1200⁰C;
 - b) pneumatically injecting a metal containing compound into said molten matrix to form an alloy melt;
 - c) injecting a hydrocarbon compound into said melt, to generate in-situ carbon for reacting with said melt, to obtain molten alloy containing metallic carbide particulate; and
 - d) casting and solidifying said molten alloy.
2. The process as claimed in claim 1, wherein said metal compound is injected into said molten matrix through a feeder attached to a submersible lance, said lance being immersed in said molten matrix.
3. The process as claimed in claim 1, wherein the hydrocarbon compound is injected into said melt through a feeder attached to a submersible lance, said lance being immersed in said molten matrix.
4. The process as claimed in claim 1, wherein said metal compound is injected along with the hydrocarbon into said molten matrix.
5. The process as claimed in claim 1, wherein the hydrocarbon compound is selected from the group consisting of paraffin and acetylene gas.

6. The process as claimed in any one of the preceding claim, wherein said metal compound and the hydrocarbon compound is injected pneumatically using pressurized carrier gas.
7. The process as claimed in claim 6, wherein the carrier gas is selected from the group consisting of argon and nitrogen.
8. The process as claimed in claim 1, wherein said metal compound is in the powder form.
9. The process as claimed in claim 1, wherein said metal compound is a titanium compound.
10. The process as claimed in claim 9, wherein the titanium compound is selected from the group consisting of potassium titanium fluoride and titanium oxide.
11. The process as claimed in claim 1, wherein said molten matrix further comprises at least one alloying element selected from the group consisting of copper, zinc, magnesium and silicon.
12. The process as claimed in claim 1, wherein the molten alloy formed in step c) is further agitated with the carrier gas for the uniform distribution of metallic carbide particulate in the molten alloy.
13. The process as claimed in claim 1, wherein said molten matrix is maintained at a temperature in the range of 850⁰C to 1000⁰C.

14. The process as claimed in claim 1, wherein the process further comprises the step of adding at least one alloying element to the molten alloy containing metallic carbide particulate, said alloying element selected from the group consisting of copper, zinc, magnesium and silicon.

15. A reinforced aluminum-metal matrix composite as claimed in claim 1, having number of defects less than 15/100micron².

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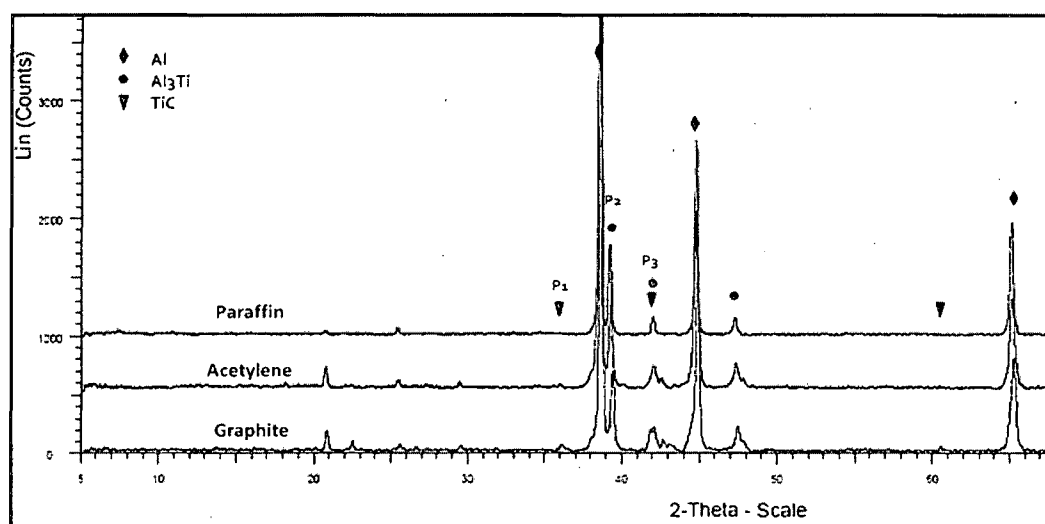


FIGURE 1

2 / 2

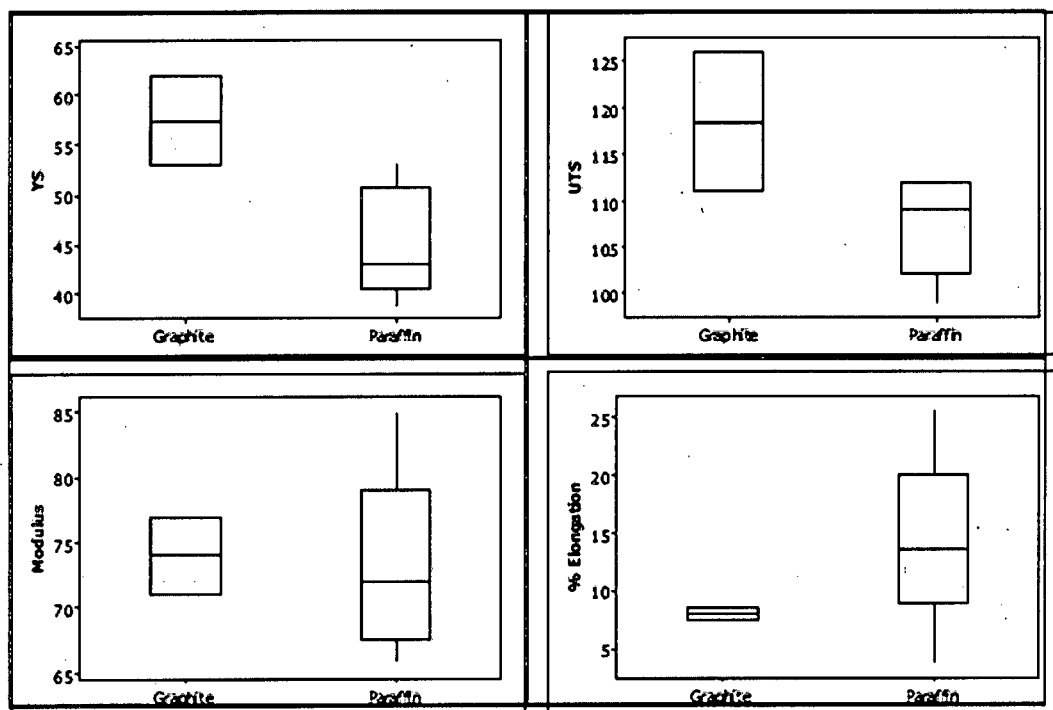


FIGURE 2