A STUDY ON THE HIGH TEMPERATURE METAL COMPOSITES OF ALUMINUM AND IRON WITH GRAPHENE OXIDE

PROJECT AT

TATA IRON AND STEEL CO.
JAMSHEDPUR

CSIR-IMMT
BHUBANESWAR

Report by:

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INDIAN INSTITUTE OF TECHNOLOGY PATNA

ACKNOWLEDGEMENT

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I am thankful to Almighty and my parents without whose constant support , this summer internship would not have been a success.

Abhijeet Singh

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ABOUT THE INDUSTRY AND INSTITUTE:

Fig 1 a: Inset image of CSIR The Indian Institute of Minerals and Materials Technology

CSIR-Institute of Minerals and Materials Technology (IMMT) was setup in 1964 as Regional Research Laboratory, Bhubaneswar in the eastern part of India under the aegis of the Council of Scientific and Industrial Research (CSIR), New Delhi The institute has expertise in conducting basic research and technology oriented programs in a wide range of subjects to ensure a sustainable growth of the mining, mineral and metals industries

Fig 1 b: Inset image of TISCO plant Jamshedpur

TATA STEEL LIMITED (FORMERLY TATA IRON AND STEEL COMPANY LIMITED (TISCO)) IS AN INDIAN MULTINATIONAL steel-making company headquartered in Mumbai, Maharashtra, India, and a subsidiary of the Tata Group. It was the 11th largest steel producing company in the world in 2013, with an annual crude steel capacity of 25.3 million tonnes, and the second largest steel company in India (measured by domestic production) with an annual capacity of 9.7 million tonnes after SAIL

ABSTRACT:

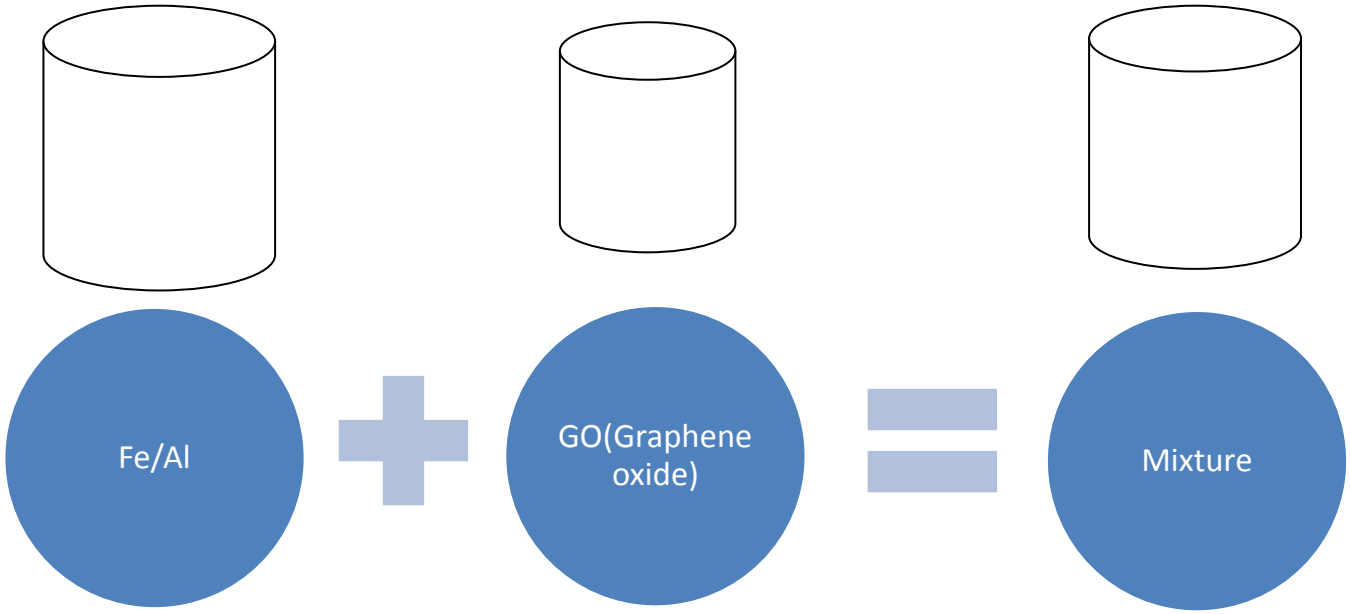
Of late, Graphene has occupied the attention of almost all researchers working globally in the area of materials science. And latest in this addition is the Graphene oxide and RGO(Reduced Graphene Oxide) although they do not share the exact same chemical and physical and chemical properties as pristine Graphene, due to some structural defects. After the pioneering work of work of Graphene, there has been considerable amount of work being done in the field of graphene oxide, the numerous pompous applications that it could serve.

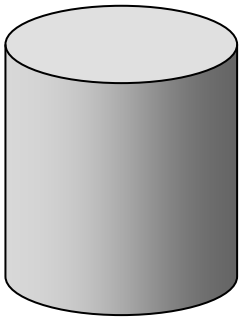
INTRODUCTION:

One such potential application that we are going to study could in the field of Metal composites. Metal composites have been so vital for human endeavours; take for example the space suit of astronauts, the materials used in the space shuttle, have to bear temperatures ranging from -50 degree Celsius to temperatures in excess of 1500 degree Celsius and this transition comes in span of seconds. No natural material can achieve that, and here is where the role of metal composites comes in.

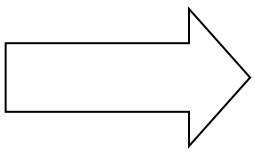
One such type of metal composites that we are to prepare and analyse is with the use of Graphene oxide. Why did we go for it? The very first question that must cross your mind is why graphene oxide? This is because graphene oxide shows similar kinds of properties to graphene –high surface area, good electrical conductivity, no rusting(as in the case with metals) and other plethora of advantages that comes with it and that too, at a cheaper price and manufacturing costs than graphene which is very difficult to prepare in great quantities. This is due to the fact that the excellent properties of graphene like thermal and electrical conductivity and structural properties *etc.* can be easily harnessed to maximum extent by developing graphene-based composites. The choice of the second material for fabrication depends on the type of application. Since, we are looking forward for the application of these composites in steel industry; we have decided to go for iron and aluminium composites for our initial tests. So, we decided to give it a try and if we found graphene oxide in metal matrix, our initial experiment could go successful and we can look forward to a new form of metal composites that may find huge number of applications in future.

EXPERIMENT PLAN:





Metal composite Ingot



Analysis:

- Powder XRD
- SEM Analysis

Fig 2 :The diagrammatic Representation of the experimental process to be carried out.

PROCEDURE:

we have decided to prepare four samples. We are going to designate them as follows:

Sample **Fe-0.5**: This sample contains 200 grams of Iron powder mixed uniformly with 1 gram of graphene oxide which is 0.5 percent of the iron taken.

Sample **Fe-1.0**: This sample contains 200 grams of Iron powder mixed uniformly with 2 gram of graphene oxide which is 1 percent of the iron taken.

Sample **Al-0.5**: This sample contains 200 grams of Aluminium powder mixed uniformly with 1 gram of graphene oxide which is 0.5 percent of the iron taken.

Sample **Al-1.0**: This sample contains 200 grams of Aluminium powder mixed uniformly with 2 gram of graphene oxide which is 1 percent of the iron taken.

The description of the procedure for each one of them is elaborated below:

Fe-0.5:

Iron powder weighted: 200.057 grams

Graphene oxide weighted: 1.003 grams

This mixture was thoroughly mixed in a beaker manually and then given for plasma treatment.

Plasma treatment for Iron Composite: Plasma treatment is procedure in which a high discharge of current is generated between two electrodes which are separated by a small distance, thereby creating an electric arc which produces very high temperatures of about 2000^{o} C resulting in formation of plasma. This all happens in an inert atmosphere of Argon thereby reducing the chances of oxide formation. For optimum results the voltage is kept around 50-60 V and current around 300 A. There is always a preheating required for the graphite crucible for approximately 2-3 minutes followed by heating of approximately 10 minutes for the iron sample. There is an approximate loss of 10-20 percent of the sample during plasma treatment which may be caused by various conformational losses of the sample the power source and the adjustments of the crucible.

(a)

(b)

Fig 3 a : The plasma reactor

Fig 3 b: The plasma reactor during operation

For the Fe-0.5

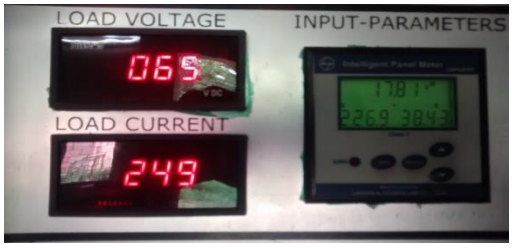
Time for which the heating has been done: 11 minutes and 22 seconds.

Initial conditions for plasma source during preheating.

Load voltage : 22 Volt Current :252 Ampere

Final conditions for plasma source .

Load voltage : 65 Volt Current : 249 Ampere



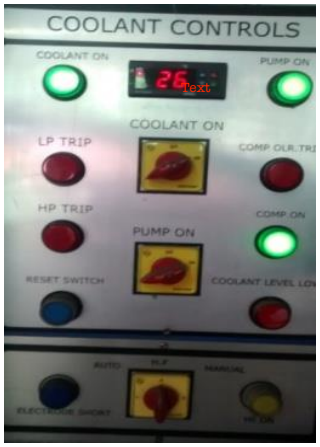


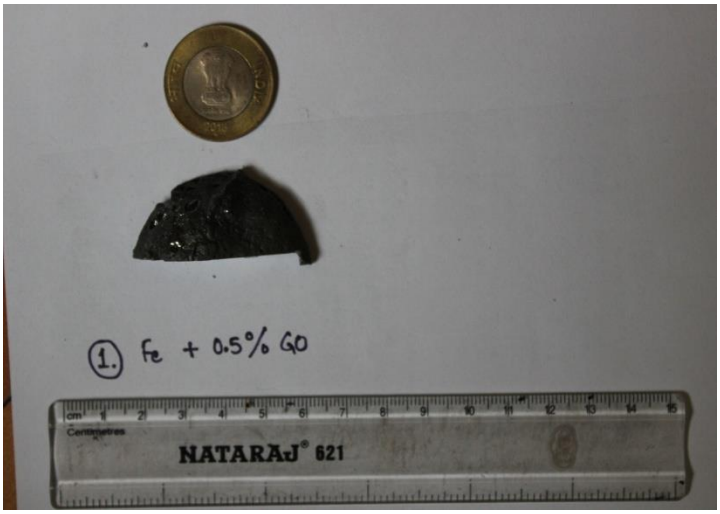
Fig 4:Snapshot of the control panels on the plasma power source during plasma discharge: these generated the temperatures in excess of 1800^{o} C resulting in the melting of the sample.

The entire procedure was covered under camera and video recording, some of the snapshots are being shown

Final observations:

The sample was observed to melt completely with the complete separation of the metal with the slag.

Fig 5: The Ingot of the iron graphene oxide metal (0.5) composite in true scale



Physical observations:

The sample was observed to be course metallic initially suggesting the sample has a carbon present in the sample.

Analysis:

Powder XRD Analysis:

The sample was conditioned to powder treatment for XRD analysis with the default conditions as stated below:

- Internal wavelengths used from anode material: Cobalt (Co)
- Original K-Alpha1 wavelength = “1.78901”
- Used K-Alpha1 wavelength = “1.78901”
- Original K-Alpha2 wavelength = “1.79290”
- Used K-Alpha2 wavelength = “1.79290”
- Original K-Beta wavelength = “1.62083”
- Used K-Beta wavelength = “1.62083”
- Irradiated length = “10.00000”
- Step axis value = “0.00000”

- Offset = “0.00000”

- Sample length = “10.00000”

Interpolate Step size:

- Step Size = “0.01”

Search Peaks:

- Minimum significance = “2”

- Minimum tip width = “0.01”

- Match intensity = “Yes”

- Identify = “Yes”

- Max. no. of accepted patterns = “5”

- Minimum score = “25”

- Min. new lines / total lines = “50”

- Search depth = “10”

- Minimum new lines = “3”

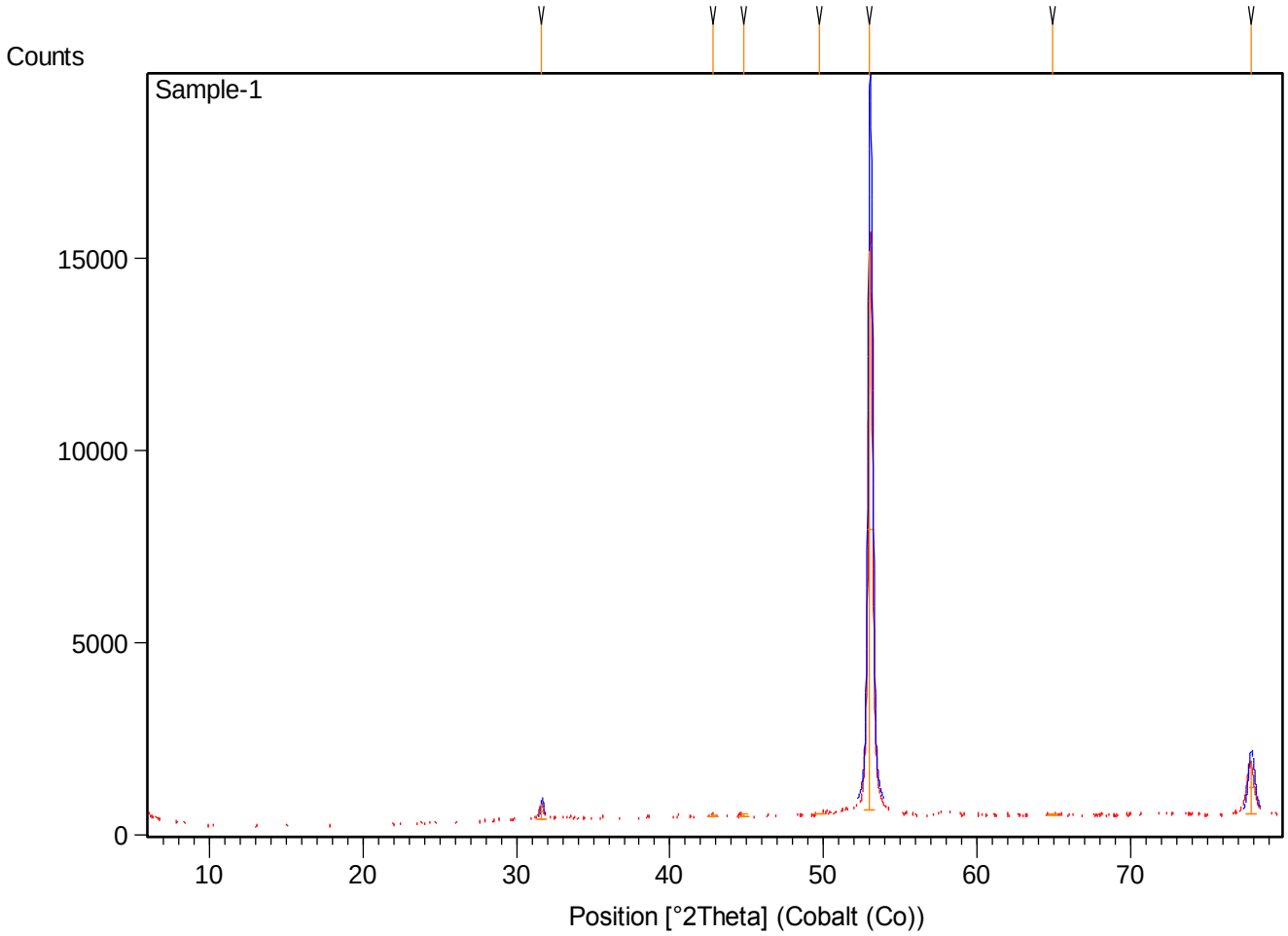
- Minimum scale factor = “0.0001”

These conditions are going to be same for each of the sample that we have tested.

XRD Data Set :

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
31.641850	381.366800	0.200736	3.28338	2.62
42.776780	76.985930	0.401472	2.45457	0.53
44.812330	107.987800	0.468384	2.34844	0.74
49.707440	55.159670	0.669120	2.12978	0.38
53.022620	14529.710000	0.284376	2.00539	100.00
64.881360	58.982060	0.669120	1.66874	0.41
77.825970	1314.601000	0.401472	1.42509	9.05

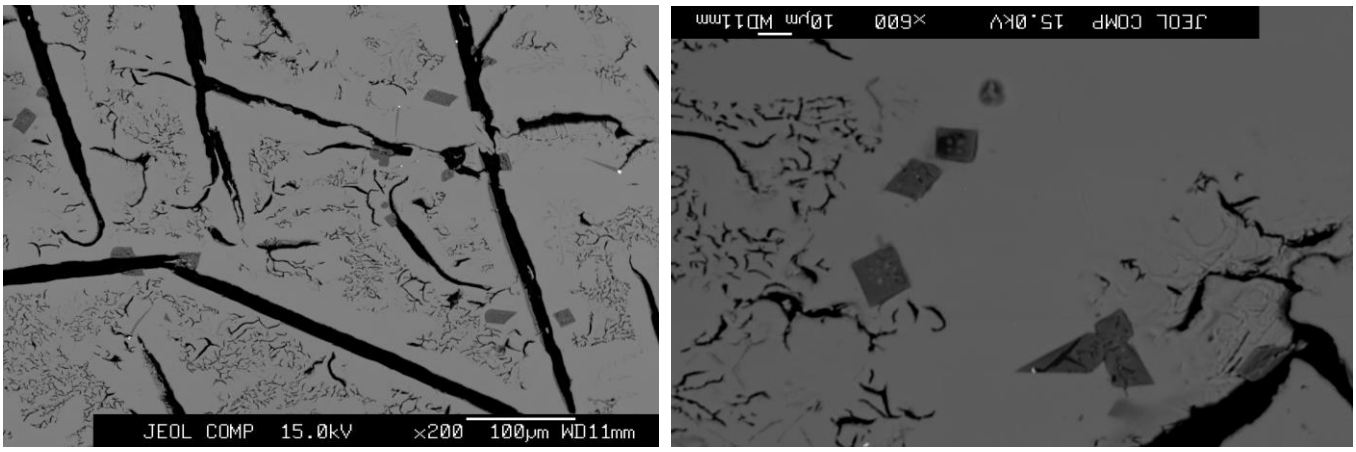
Fig 6: The XRD graph obtained for Fe 0.5 sample analysis



SEM(Scanning electron Microscope) Analysis :

The sample was further subjected to SEM Analysis :

Fig 7: The images obtained during the SEM Analysis for the Fe 0.5 sample



Fe-1.0

Iron powder weighted: 200.012 grams

Graphene oxide weighted: 2.004 grams

This mixture was thoroughly mixed in a beaker manually and then given for plasma treatment.

Time for which the heating has been done: 10 minutes and 7 seconds.

Final conditions for plasma source during operation.

Load Voltage: 67 Volt Current : 297 Ampere

Final observations:

The sample was observed to melt completely with the complete separation of the metal with the slag.

Fig 8: The Ingot of the iron graphene oxide(1.0) metal composite in true scale

Physical observations:

The sample was observed to be course metallic initially suggesting the sample has a carbon present in the sample.

Analysis:

Powder XRD Analysis:

The sample was conditioned to powder treatment for XRD analysis with the default conditions as stated above for the sample Fe-0.5

XRD Data Set :

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
49.108630	114.211900	0.301104	2.15411	13.79
51.168390	828.279400	0.234192	2.07290	100.00
60.051680	216.341800	0.535296	1.78891	26.12
78.897510	342.033000	0.401472	1.40883	41.29

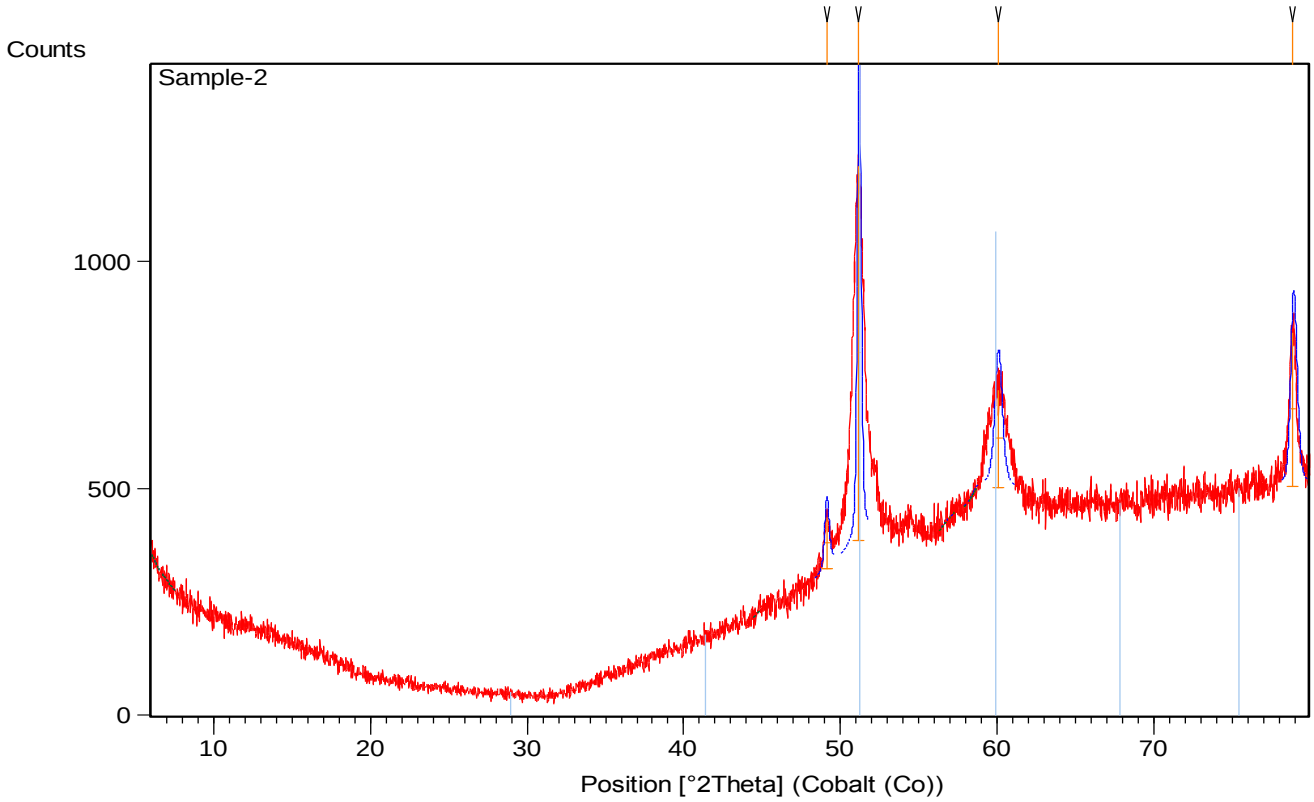
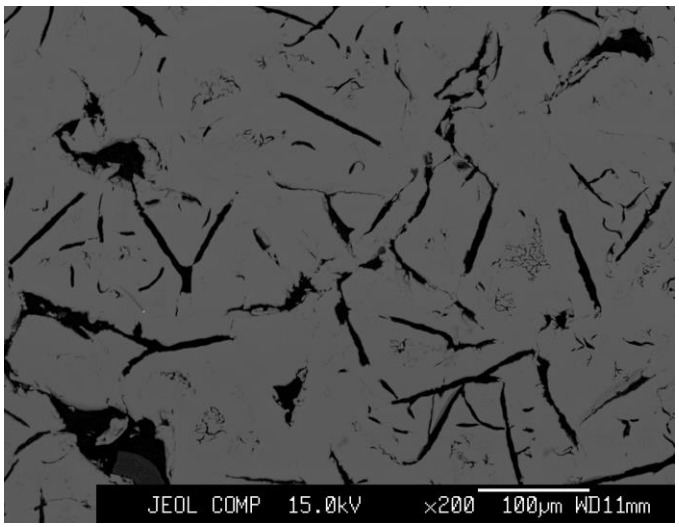


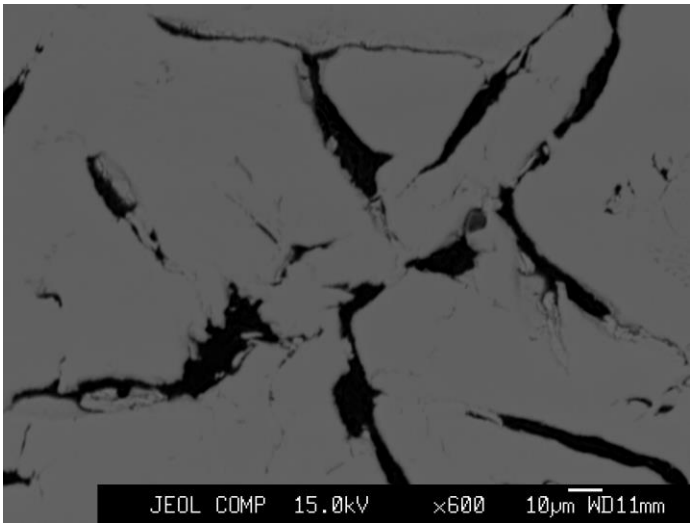
Fig 9: The XRD graph obtained for Fe 1.0 sample analysis

SEM(Scanning electron Microscope) Analysis :

The sample was further subjected to SEM Analysis :

Fig 10: The images obtained during the SEM Analysis(1.0) for the sample are shown below





Al-0.5:

Iron powder weighted: 200.006 grams

Graphene oxide weighted: 1.003 grams

This mixture was thoroughly mixed in a beaker manually and then given for plasma treatment.

Plasma treatment for Aluminium Composite: Plasma treatment is procedure in which a high discharge of current is generated between two electrodes which are separated by a small distance, thereby creating an electric arc which produces very high temperatures of about 1500^{o} C resulting in formation of plasma. This all happens in an inert atmosphere of Argon thereby reducing the chances of oxide formation. For optimum results the voltage is kept around 50-60 V and current around 250 A. There is always a preheating required for the graphite crucible for approximately 5-6 minutes followed by heating of approximately 20 minutes for the aluminium sample. There is an approximate loss of 10-20 percent of the sample during plasma treatment which may be caused by various conformational losses of the sample the power source and the adjustments of the crucible.

Time for which the heating has been done: 23 minutes and 62 seconds.

Final conditions for plasma source during operation.

Load voltage :48 Volt Current :250 Ampere

Final observations:

The sample was observed to melt completely but the separation of the metal with the slag was not observed completely.

Fig 11: The Ingot of the Aluminium graphene oxide metal (0.5) composite in true scale

Physical observations:

The sample was observed to be course metallic with slag present on the outer layer initially suggesting the sample has a carbon present in the sample.

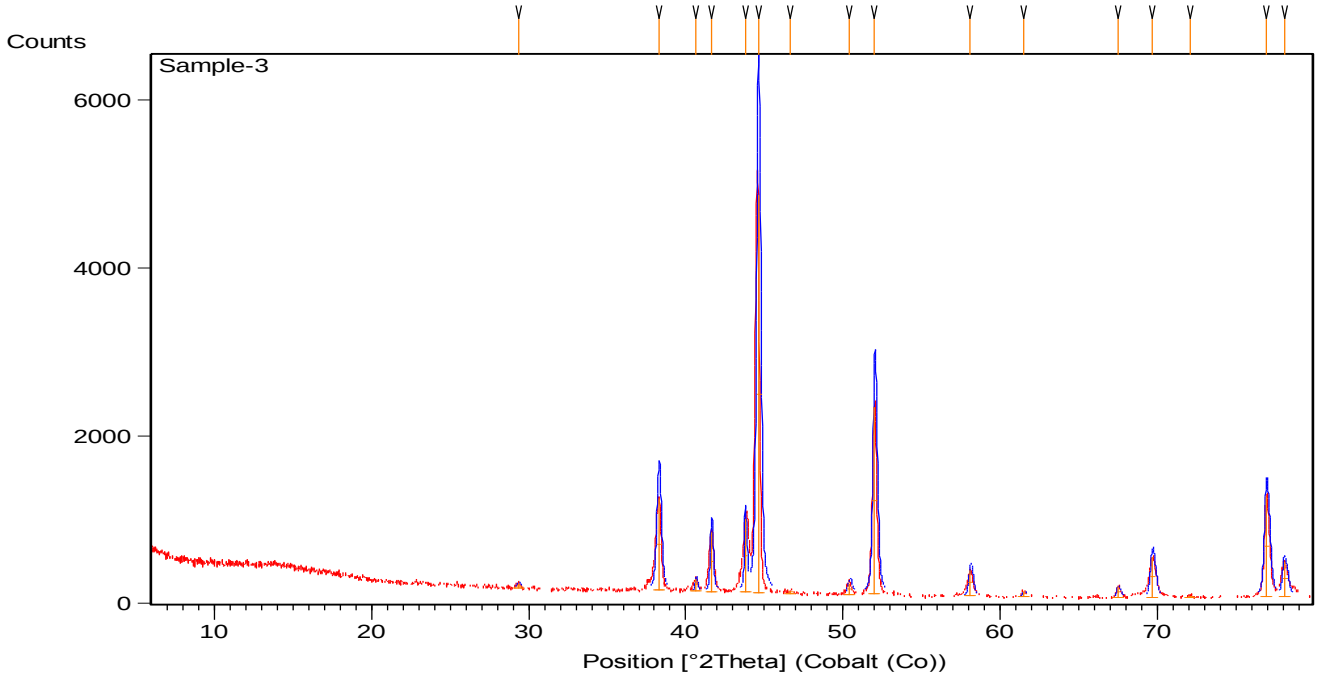
Analysis:

Powder XRD Analysis:

The sample was conditioned to powder treatment for XRD analysis with the default conditions as stated above for the sample Fe-0.5

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
29.337550	58.694530	0.267648	3.53493	1.24
38.275140	1097.773000	0.301104	2.73048	23.12
40.646380	153.234900	0.133824	2.57735	3.23
41.629810	706.474500	0.184008	2.51907	14.88
43.783000	876.139800	0.117096	2.40084	18.45
44.608860	4748.657000	0.267648	2.35860	100.00
46.626740	32.760600	0.401472	2.26186	0.69
50.412710	135.995300	0.267648	2.10189	2.86
52.018970	2233.739000	0.267648	2.04131	47.04
58.115040	309.634300	0.267648	1.84306	6.52
61.495240	72.201070	0.200736	1.75089	1.52
67.501970	135.030300	0.200736	1.61119	2.84
69.690080	487.685400	0.301104	1.56671	10.27
72.062130	19.057910	0.401472	1.52179	0.40
76.948140	1200.055000	0.301104	1.43878	25.27
78.090060	418.270800	0.267648	1.42103	8.81

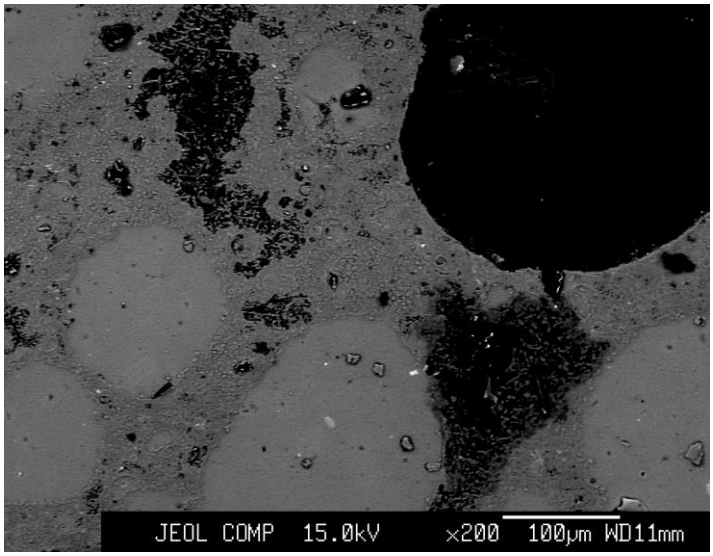
Fig 12: The XRD graph obtained for Al 0.5 sample analysis

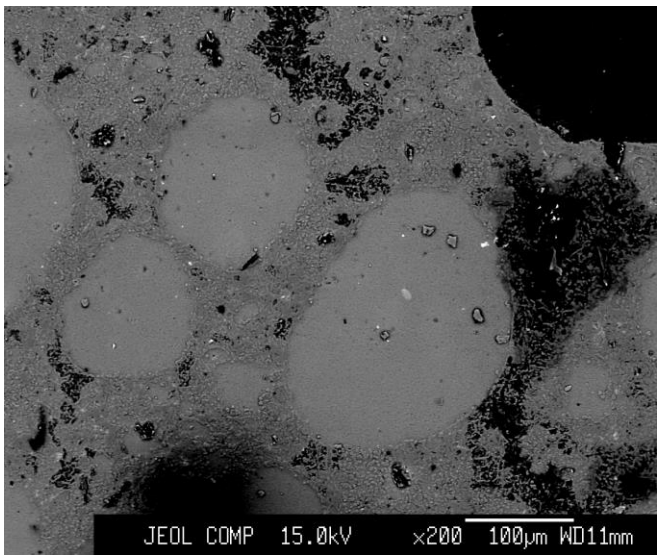


SEM(Scanning electron Microscope) Analysis :

The sample was further subjected to SEM Analysis :

Fig 13: The images obtained during the SEM Analysis for the sample (Al 0.5)





Al-1.0:

Iron powder weighted: 200.006 grams

Graphene oxide weighted: 1.003 grams

This mixture was thoroughly mixed in a beaker manually and then given for plasma treatment.

Plasma treatment for Aluminium Composite: Plasma treatment is procedure in which a high discharge of current is generated between two electrodes which are separated by a small distance, thereby creating an electric arc which produces very high temperatures of about 1500^{o} C resulting in formation of plasma. This all happens in an inert atmosphere of Argon thereby reducing the chances of oxide formation. For optimum results the voltage is kept around 50-60 V and current around 250 A. There is always a preheating required for the graphite crucible for approximately 5-6 minutes followed by heating of approximately 20 minutes for the aluminium sample. There is an approximate loss of 10-20 percent of the sample during plasma treatment which may be caused by various conformational losses of the sample the power source and the adjustments of the crucible.

Time for which the heating has been done: 23 minutes and 62 seconds.

Final conditions for plasma source during operation.

Load voltage : 69 Volt Current :250 Ampere

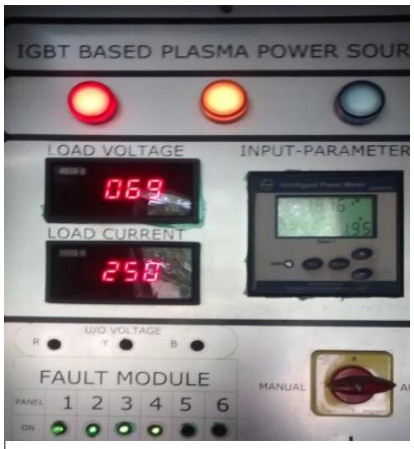


Fig 14: Snapshot of the control panel of plasma reactor during operation

Fig 15: The Ingot of the Aluminium graphene oxide metal composite (1.0) in true scale

Final observations:

The sample was observed to melt completely but the separation of the metal with the slag was not observed completely.

Physical observations:

The sample was observed to be course metallic with slag present on the outer layer initially suggesting the sample has a carbon present in the sample.

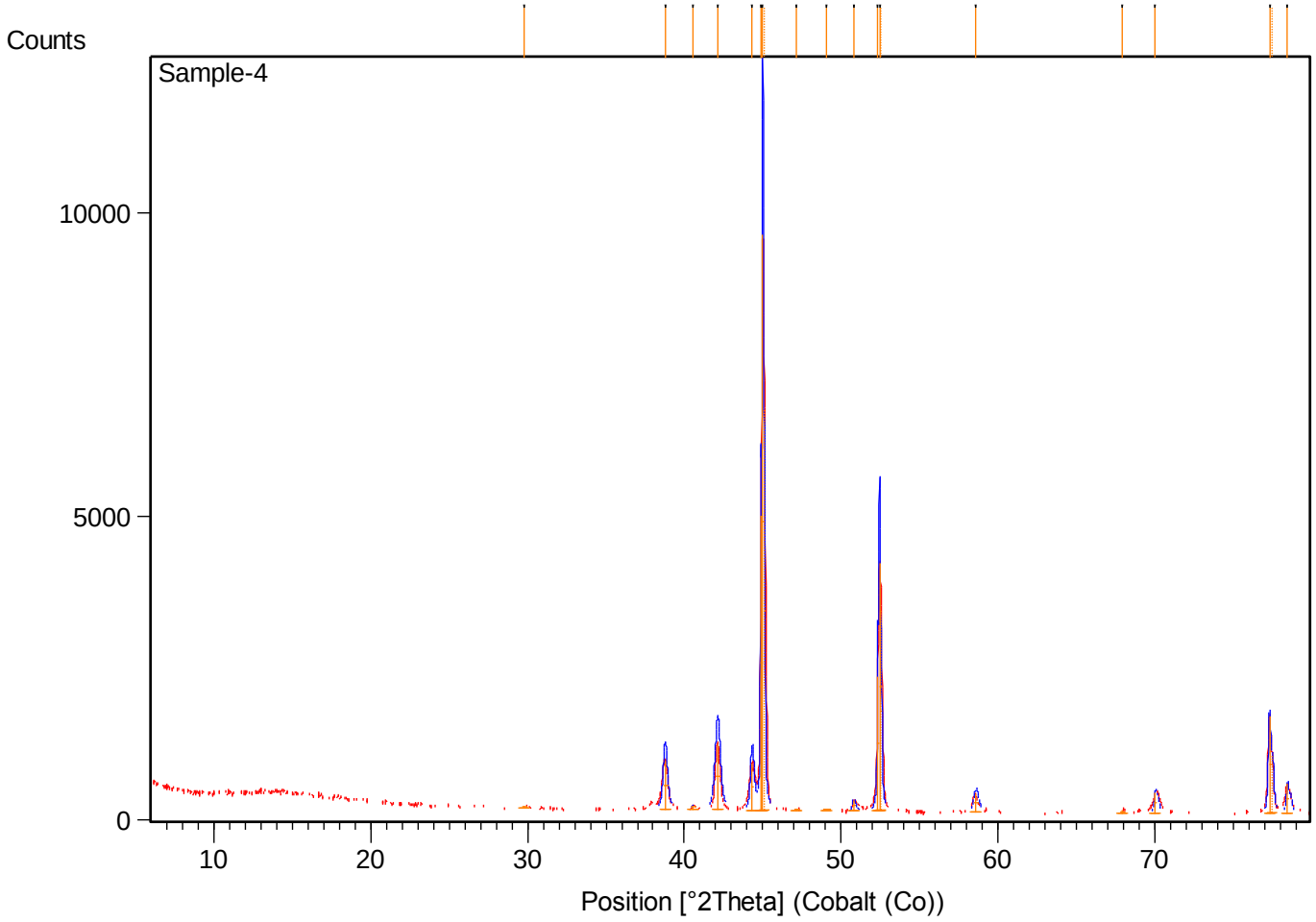
Analysis:

Powder XRD Analysis:

The sample was conditioned to powder treatment for XRD analysis with the default conditions as stated above for the sample Fe-0.5

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
29.762530	32.554920	0.401472	3.48557	0.34
38.759720	790.041300	0.301104	2.69763	8.33
40.587570	65.456240	0.267648	2.58093	0.69
42.099420	1084.400000	0.368016	2.49223	11.43
44.303270	784.356000	0.234192	2.37404	8.27
44.888690	4870.754000	0.081600	2.34295	51.34
45.005180	9487.967000	0.102000	2.33720	100.00
45.121040	6593.692000	0.102000	2.33658	69.50
47.171580	55.036070	0.326400	2.23558	0.58
49.038410	26.040700	0.489600	2.15544	0.27
50.777980	172.244200	0.204000	2.08625	1.82
52.334350	2224.198000	0.081600	2.02839	23.44
52.445050	4093.343000	0.122400	2.02442	43.14
52.554710	3285.270000	0.102000	2.02488	34.63
58.569550	307.793800	0.285600	1.82869	3.24
67.949520	60.237680	0.408000	1.60068	0.63
70.056140	349.939600	0.204000	1.55843	3.69
77.327670	1609.080000	0.183600	1.43178	16.96
77.536630	956.332700	0.081600	1.43163	10.08
78.475660	493.417500	0.183600	1.41415	5.20

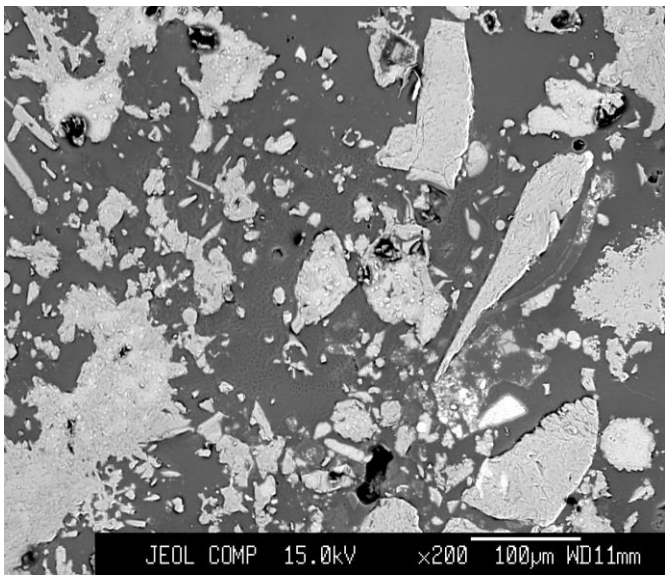
Fig 16: The XRD graph obtained for Al 1.0 sample analysis

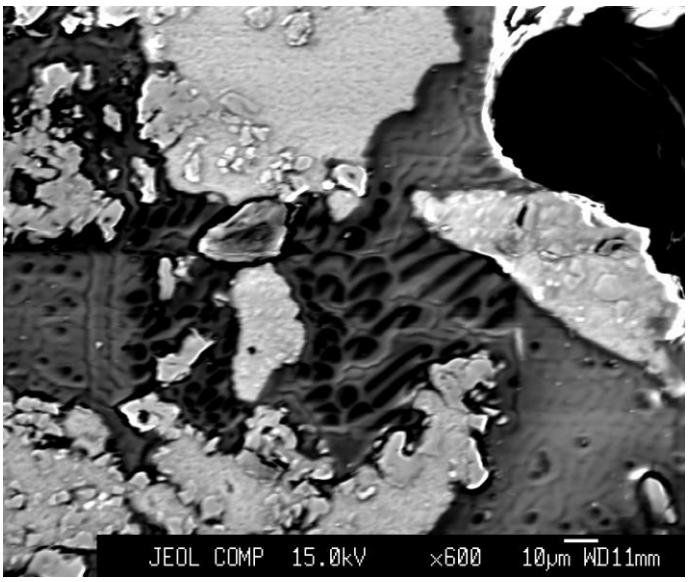


SEM(Scanning electron Microscope) Analysis :

The sample was further subjected to SEM Analysis :

Fig 17: The images obtained during the SEM Analysis for the sample Al 1.0





RESULTS AND CONCLUSIONS

The presence of light grey areas along with the dark regions of the metal composite in the SEM Analysis suggest that the graphene oxide is intermixed in the layers of metal matrix, also the indication of the presence of carbon in the Powder XRD suggest that the samples have carbon is intermixed in the metal composite.

So, this project seems to be a great success. There are several other tests that are needed to be performed on these kinds of metal composites to suggest their advantages in the steel industry sector. But there is an enormous amount of research and work that is still to be achieved and done regarding these metal composites.

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