High temperature behavior of Graphite in comparison with HSLA Steel of Aerospace Grade and Aluminum Alloys for Aerospace Application

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Certificate

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ABSTRACT

High strength and low alloy (HSLA) steel which is also known as structural steel and Aluminum Alloys such as 6xxx, 7xxx and 2xxx series has already been used for structural application in aerospace industry. They show very good mechanical properties at low temperatures but their strength start to decrease at high temperature. To increase the service life at elevated temperature, there is required a material which can hold at elevated temperature and its property do not decrease such as HSLA steel and Aluminum Alloys, for such requirement, C-C composite (graphite) system in combination with HSLA Steel and Aluminum Alloys can be used which can bear high temperature and also show very good specific strength at elevated temperatures. This project includes the study and investigation of behavior of the three systems of material (HSLA steel, Aluminum alloys and C-C composite (graphite)) and comparing high temperature behavior and properties of C-C composite (graphite) with the HSLA steel and aluminum alloys system.

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CHAPTER 1

INTRODUCTION

1.1. Background:

Different materials have been used as structural material. In these materials, HSLA steel and aluminum alloys have prominent position due their high strength, toughness, and high fatigue strength and corrosion resistance properties. Both materials have unique properties and have different applications as well. These application include wings and aircraft frames, structure of Missiles (ballistic and Cruise), space shuttles, Engine parts and Turbine blades etc. There is advancement in materials with the passage of time so new systems are introducing which can also be act as structural materials while giving an advantage of having high specific strength and ease of manufacturability.

1.2. High Strength Low Alloy steel:

High strength low alloy (HSLA) steels are used for structural application and often known as structural steel which contain minute quantity of alloying elements, such as vanadium, niobium, , copper, chromium, titanium, zirconium, magnesium, molybdenum and aluminum etc which provide strength through the formation of carbide, carbonitrides and nitride and increasing the hardenability of the steel. Each alloying element provide its own special property for example Magnesium up to 2% provide better formability and weldability and also increases the hardenability of the steel. Similarly, it contains very minute quantity of niobium (approx. 0.05%), vanadium (approx. 0.1%), and titanium (approx. 0.07%) to provide strengthening purpose through grain refinement and precipitation hardening.

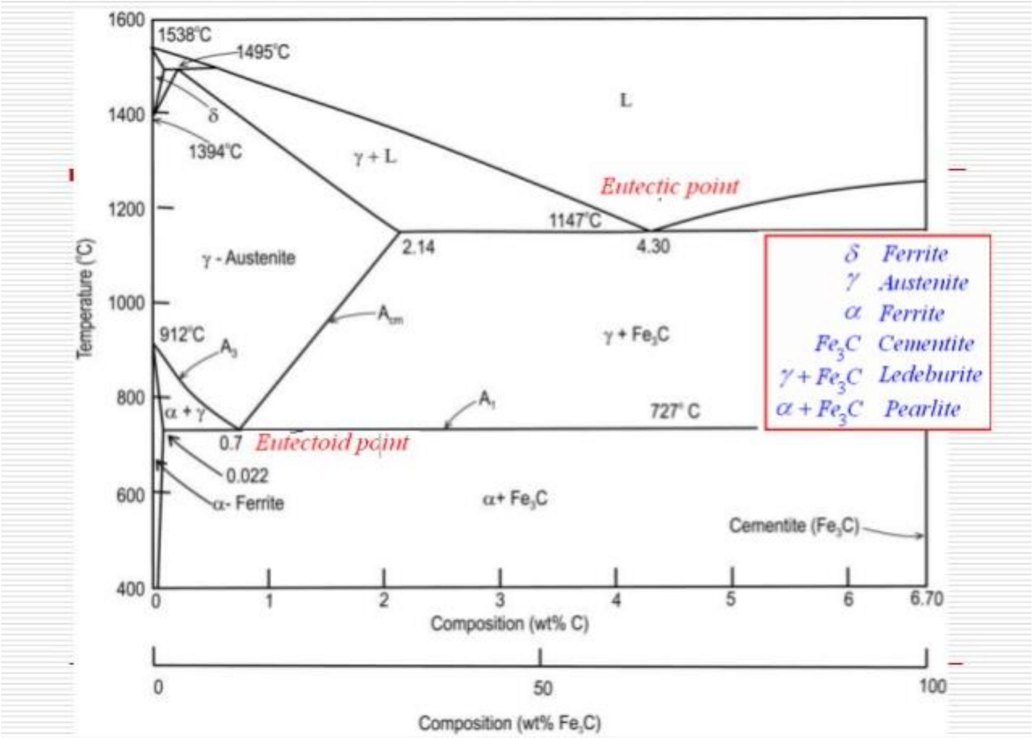

Figure 1.1: Iron Carbon Phase Diagram.

HSLA steel contain carbon contents ranging from approx. 0.05%-0.25% and total alloying content is less that 2%, due to this reason they are also known as micro-alloyed steel. HSLA steel provide better mechanical properties and better corrosion resistance.

On the basis of applications and microstructure formed in final product, HSLA steel can be divided into six types:

1.2.1. Wheathering steel:

It contains small amount of copper and phosphorous, which provide corrosion resistance and solid solution strengthening. We know that copper and phosphorus both form solid solution at elevated temperature. Due to corrosion resistance property, it is used for steel structures which are exposed to the open atmosphere. Such as building and bridge construction. [1]

1.2.2. Micro-alloyed ferrite and pearlite steels:

As the name suggest, this type contain ferrite and pearlite microstructure in the end product. Such micro structure is obtained by adding very small amount of strong carbide and nitride formers like vanadium, titanium and niobium which helps in, precipitation strengthening and possibly, control on transformation temperature and grain refinement. [1]

1.2.3. As-rolled Pearlite steel:

It has pearlite structure and contain manganese with small addition of other alloying element to enhance strength toughness, formability and weldability. It forms globular bainite along with ferrite due to addition of manganese. The ductility enhance due to formation of globular bainite which helps in dislocation movement. [1]

1.2.4. Acicular ferrite steels:

It is low carbon bainite steels, it contain carbon content less than 0.05%. These steels provide high yield strength upto 690 MPa along with excellent combination formability, good toughness and weldability. Acicular ferrite steel are obtained when we transform steel through bainitic nose, hence it is also called low carbon bainite steel. The carbon diffuses in the non-diffusional phase (Martensite) and forms feathery microstructure which is Bainite. These structure increases the strength of the steel and increases the hardness. [1]

1.2.5. Dual Phase steels:

It contain two phases, martensite and ferrite. The microstructure of martensite inserts dispersed in ferritic matrix due to which we obtain excellent combination of ductility and high tensile strength. Due to faster cooling rate some of the austenite don’t get enough time to transform into ferrite and hence changes into martensite which is then dispersed in the ferrite matrix. It can be recognized by the needle like lath structure form by martensite, the grain of martensite contain Ɛ-carbon needles which increases the hardness of the material. [1]

1.2.6. Inclusion Shaped Controlled steel:

In this steel, small amount of calcium, zirconium, titanium or rare earth metals are added which changes the shape of the sulphide inclusion in spherical globules which was in elongated stringers. Which increases the ductility and toughness of the material. [1]

1.3. Applications:

HSLA steels have vast variety of application, it is not limited to few fields. It has wide range due to its special properties these are used from simple Car components to the advanced technology air crafts. Applications for HSLA steel are as follows:

1. Oil and gas pipeline
2. Heavy duty highway and off road vehicles: Trucks structure
3. Aerospace applications: Rocket motor Casing, Aircraft structure.
4. Industrial equipment, storage tanks
5. Mine and Railroad cars
6. Passenger car components: Car Chassis, Crank shaft.
7. Bridges, offshore structure
8. Power Transmission towers
9. Building Beams and panels.

The main focus is in aerospace industry where HSLA steel are used as structural material due to its excellent load bearing and mechanical properties. In Aerospace industry, it is mainly used in rocket motor casing, missile (ballistic and cruise) structural frame and Aircraft frames

1.4. ASTM 4240 Steel:

HSLA Steel is structural steel which has been used for structural application in aerospace industry. It is used for the structural frame of Rocket motor casing and missile frame and also it is used for making many parts of aircrafts. This material give excellent mechanical properties at low temperature but it do not perform well at high temperatures. The chemical composition, its state and mechanical properties has been given in the Table1.1.

Table 1.1: Mechanical Properties of ASTM 4240

Material	**ASTM 4240**				
Chemical Composition	**Condition**	**Mechanical Properties**			
		YS MPA	**UTS MPA**	**Elongation%**	**Hardness HB**
0.27-0.33%C 0.7-1.0%Mn ≤0.013%P ≤0.01%S 1.4-1.76%Si ≤0.25%Ni 1-1.3%Cr 0.4-0.55%Mo 0.08-0.15%V ≤0.25%Cu Balance Fe	Annealed	≥500	≥750	≥10	≥200
	Tempered	≥1325	≥1600	≥8	≥445 Or 40HRC
	Tempered	≥1100	≥900	≥8	36HRC

1.5. Aluminum Alloys:

Aluminum and its alloys have vast range of applications in industries because aluminum is an abundant element present in earth crust. Its application ranges from kitchen utensils to aircrafts. It is possible because large variety of aluminum alloys can be made by different alloying addition. Alloying elements are added in aluminum in order to increase its strength and other properties. Two most common methods are used for increasing the strength of aluminum alloys, these are as follows:

- By dispersing alloying elements to form solid solution and cold working the alloy.

- By dissolving the alloying material to form solid solution and then precipitating them in the form coherent particles.

From all metals nine elements show solid solubility which is greater than 1 % by weight and have substantially show low solubility at low temperatures. From these nine elements, only 5 elements are used because other three are expensive, these expensive elements are silver, gallium and germanium. Other 5 elements left which are being used in alloying addition are copper, magnesium, zinc, manganese and silicon. These alloying addition forms seven series of aluminum alloys, each series have a major constituent composition of element which is denoted accordingly for example in 7xxx series major constituent element is zinc. Other series are as follows:

- 1xxx series are the pure aluminum which contain approx. 99% aluminum composition and these series have ability to be work hardened.
- 2xxx series have major alloying element of copper. We can increase strength of this series by precipitation hardness which comparable with the steel. They were used as the most common aerospace alloy but due to the problem of stress corrosion cracking, it is replaced by the 7xxx series in new applications.
- 3xxx series have manganese as major alloying element, and it can be work hardened to give strength.
- 4xxx series have major alloying element silicon. Silicon in aluminum increases its ease of casting, because silicon in metals increases the fluidity of the metal and decreases the melting temperature of the metal.
- 5xxx series have magnesium as major alloying element which offer excellent corrosion resistance due which it is suitable for application where corrosion resistance is required such as marine application. Most 5xxx have manganese along with magnesium.
- 6xxx series have major alloying elements magnesium and silicon both. This series are machine able, have good weldability, and strength can be increased by precipitation hardening, but its strength cannot be increased as we can increase in case of 2xxx and 7xxx series. 6061 alloy is generally used in vas applications of 6xxx series.

- 7xxx series contain zinc as major alloying element, and its strength can be increased by precipitation hardening. This series can achieve strength that no other aluminum series can achieve with strength approx. 710 MPa for 7068 alloy. Most 7xxx series alloys contain copper and magnesium also.

For aerospace applications, 7xxx, 2xxx and 6xxx series used. For structural applications aluminum 6061 has a prominent position in aerospace industry. The major application of aluminum 6061 as structural material is body frame of the Missile bodies and aircrafts. Therefore, it is our main interest to study this at high temperature.

1.5.1. Aluminum 6061:

Aluminum 6061 provide excellent joining characteristics. It has relatively high strength and high corrosion resistance along with better workability. It is widely available in market and provide excellent properties at low temperatures. It has tensile strength of 310 MPa at room temperature and have hardness value of HRB 60. Its melting point is 650°C. Due to its low melting point this material fails high temperature applications which is above 500°C. Its strength decreases as temperature rises and at the end it is completely melt or fails at higher temperature.

Aluminum 6061 have major alloying element magnesium and silicon which are present in the range of 0.8-1.2% and 0.4-0.8%. Other alloying elements present are shown in the Table 1.2:

Table 1.2: Chemical Composition of Aluminum Alloy

Element	Composition %
Al	95.7-98.5
Cr	0.041-0.3
Cu	0.15-0.41
Si	0.40-0.80
Mn	Max 0.15
Mg	0.80-1.2
Fe	Max 0.71
Zn	Max 0.25
Ti	Max 0.15

Its application include aircraft fittings, couplings, marines fittings and hardware, brake pistons, hydraulic pistons, bike frames, Rocket and missile frames etc.

1.6. Graphite:

Graphite is the allotropic form of Carbon and has shinny and slippery surface. Graphite is thermodynamically stable form of carbon at atmospheric pressure. We can say that graphite is the metastable form of carbon and it transforms into diamond at temperature above 1500°C.

The structure of graphite contains layers of carbon atoms linked hexagonally with each other, these layers are parallel to the basal plane. Due to this hexagonal structure most of the properties of graphite are anisotropic, for example, electrical conductance of graphite is anisotropic and it shows conductance in one direction. [1]

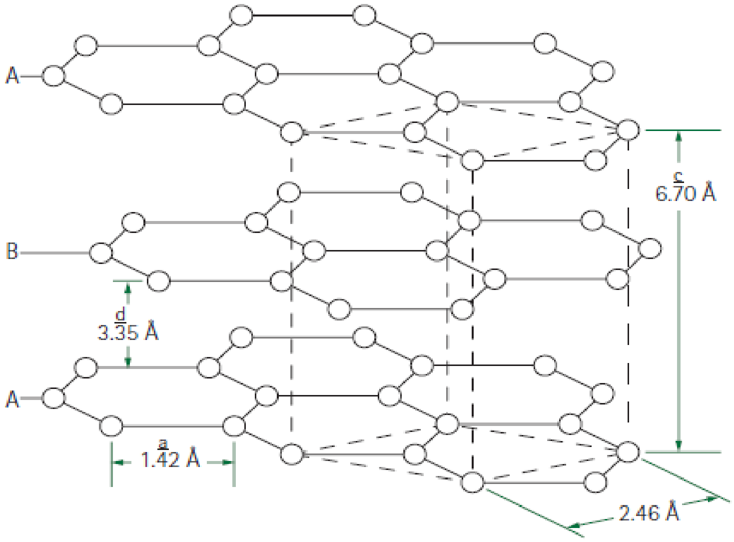

Figure 1.2: Crystal structure of graphite.

Moreover, this layered structure have weak van der wall forces between them have weak forces between them due to which:

- The fracture occur along the planes of the graphite.
- The interstitial compounds are formed.
- It provides compressive and lubricating properties along with many other.

The graphite shows many properties which makes it suitable for high temperature properties. Graphite are available in different range of density, from 1.31 g/cm3 to 1.89 g/cm3. The density plays a major part in properties of the material, as the density of the graphite increases it also increases its mechanical and physical properties.

There is little work on pure graphite for the application in aerospace industry therefore there is very little data present on high temperature application of graphite. Apart from this graphite have very good physical and mechanical properties.

Graphite have high coefficient of thermal expansion compared to steels and aluminum. The coefficient of thermal expansion of graphite ranges from 7.0x10^{-6}- 9.0x10^{-6}/°C depending on the grade of the graphite.

Moreover, thermal conductivity is greatly affected by the change in temperature, as the temperature increases it starts to decrease, thermal conductivity start to decreases around 28°C and continue to decrease as the temperature reach 3228°C. Other mechanical properties are, it has high hardness, moderate tensile strength and high wear resistance.

1.6.1. Carbon-Carbon Composite:

Carbon-carbon composite or more precisely if we say, carbon fiber reinforced carbon composites, consist of synthetic pure elemental carbon. As a solid, Carbon is very unique substance as it can be used to form many controversial structures and give very unique properties. It forms wide range as some carbon are soft and ductile while some are extremely hard and brittle. These variations in properties are not come from the alloying elements just like metals in which we can change properties by adding alloying elements, in Carbon these variation comes from the geometry, defects and number of carbon phases.

We can say that C-C composites is the intermediate synthetic graphite material, i.e. pyrolytic graphic, and carbon fiber reinforced polymers, which are the future candidate material for aerospace industry to form lightweight aircraft. Because carbon- carbon composites have the properties which are intermediate between each extreme groups of the material which have been using in aerospace industry.

C-C composites are lighter than aluminum alloys, stiffer that titanium and stronger than steel moreover, they offer high corrosion resistance even at high temperatures and also its strength do not decrease at high temperatures even at 2300°C, which steel and aluminum alloys cannot bear. It has already been used in rocket nozzles and rocket exhaust cones where temperature reaches to >1500°C. Due to this reason, we are considering C-C composite as a candidate for structural application at high temperatures at which HSLA steel and aluminum alloys become fails. [1]

1.7. Problem Statement:

HSLA steel (D406A) and aluminum alloys (6061) have structural applications in various aerospace, automotive and many other structural application where high strength of material is required. These material show very good properties at low temperature but their strength decreases with increase in temperature. For aerospace applications, we know that aircrafts and space vehicles bear high temperature because of air friction and many other factors. In Aircrafts skin temperature can reach up to 500-800°C. Therefore, such material fails sometimes at such high temperatures.

HSLA steel (D406A) has already been used for rocket motor casing and missiles frame. The main problem with HSLA steel which has seen is that it is heavy material which reduces the efficiency of aircraft and also it cannot bear high temperature exposure and its strength decrease with temperature which affects the service life. At high temperature different phenomena’s start to occur in HSLA steel due to which strength of the steel starts to decrease. These phenomena’s include decarburization, diffusion and solid state reaction started and phase transformation occurs.

Similarly, Aluminum Alloy (6061) has been used as structural material in aerospace industry. Its application include, aircraft fins, aircraft wings, missile frames etc. Apart from its light weight due to low density, Aluminum 6061 have very low melting point i.e. 650 °C, so, for structural application it cannot bear high temperature and may lose its strength or ultimately melt above 650 °C so it will completely fail at load bearing application.

So, the problem is we require a material which is light weight and have good mechanical properties, in other words, it may have good specific strength along with high temperature stability so that it can bear loads at high temperature structural application.

1.8. Objective:

As mentioned above HSLA Steel and Aluminum Alloys fails structural application at high temperature because their mechanical properties decreases at high temperature due to different phenomena’s start occurring at high temperature. Our objective is to investigate a material system (Graphite) which can sustain load bearing application at high temperature and introducing such material which has high specific strength as compare to HSLA and Aluminum alloys at high temperatures.

There is very few work and data present on graphite which can show that it can be used for high temperature application. Graphite has applications for high temperature just like rocket nose, Moderators in nuclear reactors but these are not structural applications.

Therefore, our objective is to perform high temperature testing and characterization for material analysis on HSLA steel, Aluminum Alloys and C-C composite (graphite system) to investigate their strength and behavior at high temperature and comparing graphite system with HSLA steel and Aluminum alloys for high temperature application in aerospace industry.

CHAPTER 2

LITERATURE REVIEW

In aerospace industry, advancement in material is occurring rapidly due to high speed and high strength requirement of the air crafts. As speed and altitude of the aircraft increases the temperature of the aircraft will increase automatically, because high speed requirement is fulfilled by Jet engine, more the speed more powerful will be the engine and more temperature will be raised in the engine and also speed also increase friction which also increases stress and temperature on structure of aircraft therefore to compensate this requirement aerospace industry need such material which will compensate such high temperature along with high strength.

2.1. Material Failure at high temperature:

Materials fails at high temperature because at high temperature material become thermodynamically unstable and become chemically reactive so different phenomena's start occurring in the material which leads to the material failure or decrease in strength. [1]

Phenomena's which occur at high temperature are as follows.

- Melting and softening
- Corrosion and Chemical reaction with atmosphere
- Diffusion
- Solid-Solid Phase transformation

2.1.1. Melting and softening:

Some materials have high melting point but they soften on heating at high temperature and acts as viscoelastic which obviously dangerous condition for structural application. Similarly, when metal at high temperature material start to soften or act as partial melt, it becomes more reactive with its environment because it becomes chemically unstable and material failure could occur. Impurities in the material can also be disastrous as it decreases the melting temperature therefore one requirement is that material should be as pure as possible. [1]

2.1.2. Corrosion and Chemical reaction:

As mentioned above, at high temperature the material becomes chemically unstable and it becomes more reactive with its atmosphere. These high temprature reaction include oxidation, nitration. The reaction can decrease the mechanical properties of the material as the oxidation of the material could occur or in some cases decarburization could occur which decreases the mechanical property of the material which could lead to the material failure [1]

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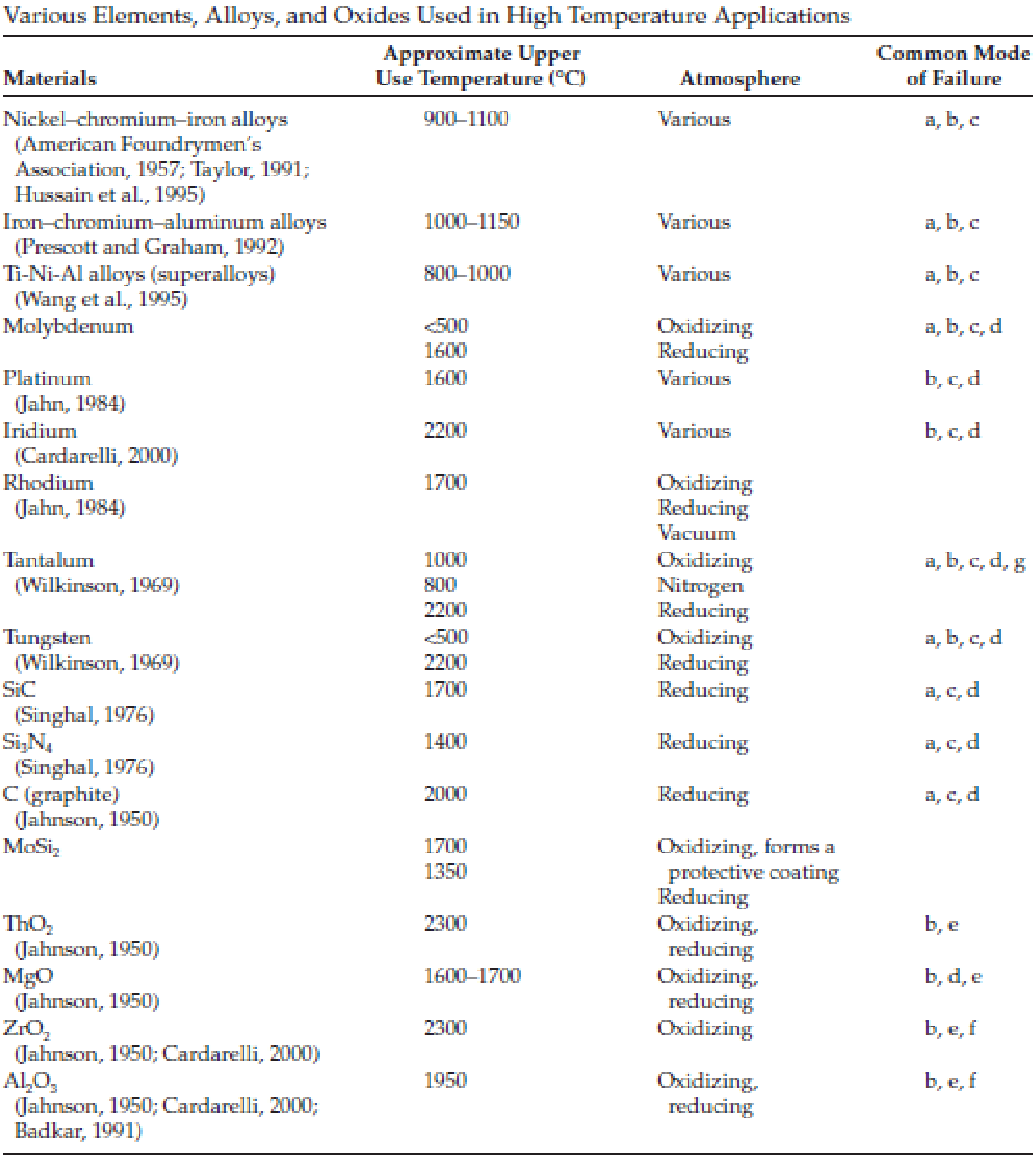
2.1.3. Diffusion:

At high temperature the grain boundaries start to grow and diffusion of alloying elements increases. The grain boundaries also start to diffuse due to which the material mechanical properties starts to decrease. In some cases, where two different materials are in contact, they can diffuse and form reacted region, this case is only feasible if such reaction is thermodynamically stable. But in structural application just like in HSLA steel, the diffusion causes microstructural changes which decreases the mechanical properties of the material at high temperature. [1]

2.1.4. Solid-Solid Phase transformation:

Sometimes, at high temperature phase transformation also occur because many materials have phase transition among different crystal structure. This phase transformation can change the mechanical properties of the material because the properties of the phase which is at high temperature could be different from that phase which is present at lower temperature. Moreover, if this phase transformation produce large volume change then it could cause crack propagation or disintegration. [1]

Various materials have been used for high temperature application such materials have different phenomena's at different temperature. A comparison is given in the Figure 2.1:



Various Elements, Alloys, and Oxides Used in High Temperature Applications

Materials	Approximate Upper Use Temperature (°C)	Atmosphere	Common Mode of Failure
Nickel–chromium–iron alloys (American Foundrymen's Association, 1957; Taylor, 1991; Hussain et al., 1995)	900–1100	Various	a, b, c
Iron–chromium–aluminum alloys (Prescott and Graham, 1992)	1000–1150	Various	a, b, c
Ti-Ni-Al alloys (superalloys) (Wang et al., 1995)	800–1000	Various	a, b, c
Molybdenum	<500 1600	Oxidizing Reducing	a, b, c, d
Platinum (Jahn, 1984)	1600	Various	b, c, d
Iridium (Cardarelli, 2000)	2200	Various	b, c, d
Rhodium (Jahn, 1984)	1700	Oxidizing Reducing Vacuum	
Tantalum (Wilkinson, 1969)	1000 800 2200	Oxidizing Nitrogen Reducing	a, b, c, d, g
Tungsten (Wilkinson, 1969)	<500 2200	Oxidizing Reducing	a, b, c, d
SiC (Singhal, 1976)	1700	Reducing	a, c, d
Si_3N_4 (Singhal, 1976)	1400	Reducing	a, c, d
C (graphite) (Jahnson, 1950)	2000	Reducing	a, c, d
$MoSi_2$	1700 1350	Oxidizing, forms a protective coating Reducing	
ThO_2 (Jahnson, 1950)	2300	Oxidizing, reducing	b, e
MgO (Jahnson, 1950)	1600–1700	Oxidizing, reducing	b, d, e
ZrO_2 (Jahnson, 1950; Cardarelli, 2000)	2300	Oxidizing	b, e, f
Al_2O_3 (Jahnson, 1950; Cardarelli, 2000; Badkar, 1991)	1950	Oxidizing, reducing	b, e, f

Figure 2.1: Application and various Elements, Alloys and Oxide at high temperature

a. Oxidation.
b. Grain growth.
c. Temperature lowered if corrosive gases or various incompatible metals oxides are present.
d. Vaporization.
e. Melting.
f. Phase transformation a problem, “stabilized zirconia avoids this but may have somewhat lower temperature limit.
g. Forms nitrides.

2.2. High Strength Low Alloy Steel:

Structural steels are engineered to provide better mechanical properties or better corrosion resistance for structural application than conventional carbon steels in normal cases because they are designed to meet specific mechanical properties rather than a chemical composition.

Mechanical properties that a material possess include:

- Elasticity
- Ultimate Tensile Strength
- Hardness
- Creep resistance
- Yield Strength
- Ductility
- Toughness
- Fatigue Strength

Structural steels show good mechanical properties as compare to other alloyed steels because of low alloy and Carbon composition. These properties are good at room temperature but as other materials, structural steels also show variation in mechanical properties as the temperature of the material is increased. With the rise in temperature the mechanical properties of the material start to decrease and at the end material fails. Generally with increase in temperature stress strain relationship affected which results in,

- Elasticity increase

- Tensile strength decrease
- Hardness decrease
- Yield strength decrease
- Ductility Increase
- Toughness increase
- Fatigue strength decrease
- Creep resistance decrease.

With increase in temperature there is change in the stress strain curve of the material from which we predict the change in the mechanical properties.

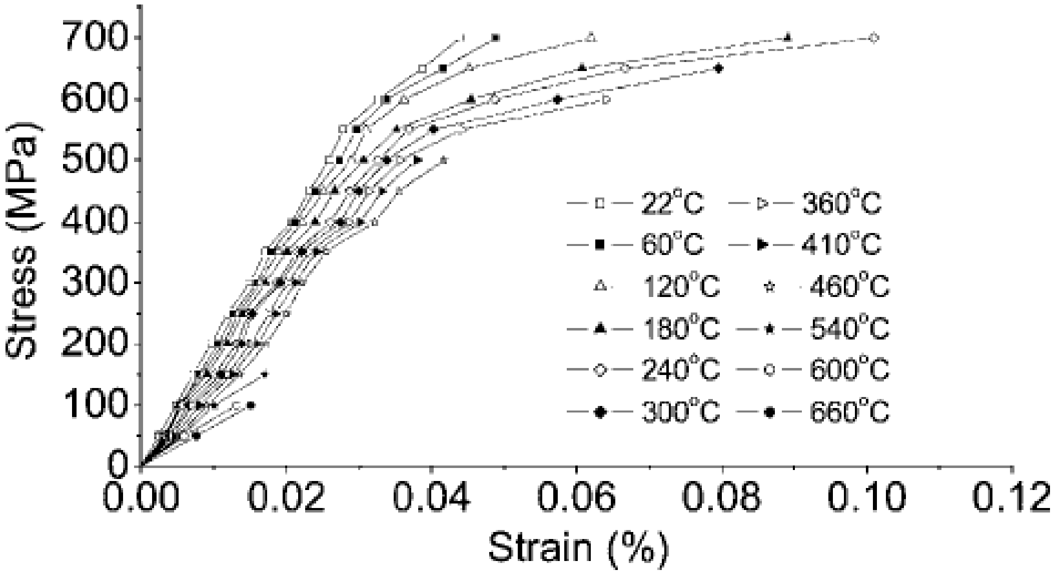


Figure 2.2: Stress Strain Curves for BISPLATE 80 at Different Temprature Ranges

Above figure 2.2, is the stress-strain curve of the BISPLATE 80 (approximately equal to ASTM A514) Structural Steel [2], from this we can see that how with increase in temperature the properties of the material changes. At 60 °C the ultimate tensile strength of the material is 650 MPa as the temperature reaches 600 °C it reduces to 100 MPa. The reason is that when temperature increases the material can easily flow with small amount of stress and large strain is produced which makes the material deform plastically and eventually failure occur. Similarly other properties also affected by temperature change.

2.2.1. Microstructure Evolution at High Temperature:

We know that when we increase the temperature of the steel, the phase transformation from ferrite and pearlite or Bainite or Martensite to austenite starts when temperature cross Eutectoid temperature i.e. 723 °C and on further heating it changes into austenite completely when it reaches in the austenite region. This is the normal case in which due to heating the system become unstable and energy of the system increases due to which it starts to transform into stable phase.

But if we apply some strain or produce some deformation at high temperatures then the microstructure transformation of deformed austenite is different from the normal condition. With the increasing of deformation amount, we obtain more needle like (lath-shaped) microstructure and less granulous microstructure. The compressive deformation effectively prevent the precipitation of carbides. Larger deformation amount or lower heating rate is conducive to the atomic diffusion, which led to the microstructure uniformity and hardness decreasing. Deformation amount and deformation temperature affects the recrystallization process of the material. [3]

Similarly, heating rate also affects the microstructure as it is important in austenite homogenization and carbide dissolution. If proper heating rate are not applied to the steel then the austenite don’t homogenize which will affect in the grain size and phase transformation.

In order to understand this behavior of microstructures at higher temperatures, let us consider an example in which an HSLA steel Q690 sample is tested at higher temperature of 1200 °C under different deformation conditions. In start, before heating the microstructure of the material is almost bainitic and have high hardness due to bainitic structure. The microstructure is shown in the figure.2.3. [3]

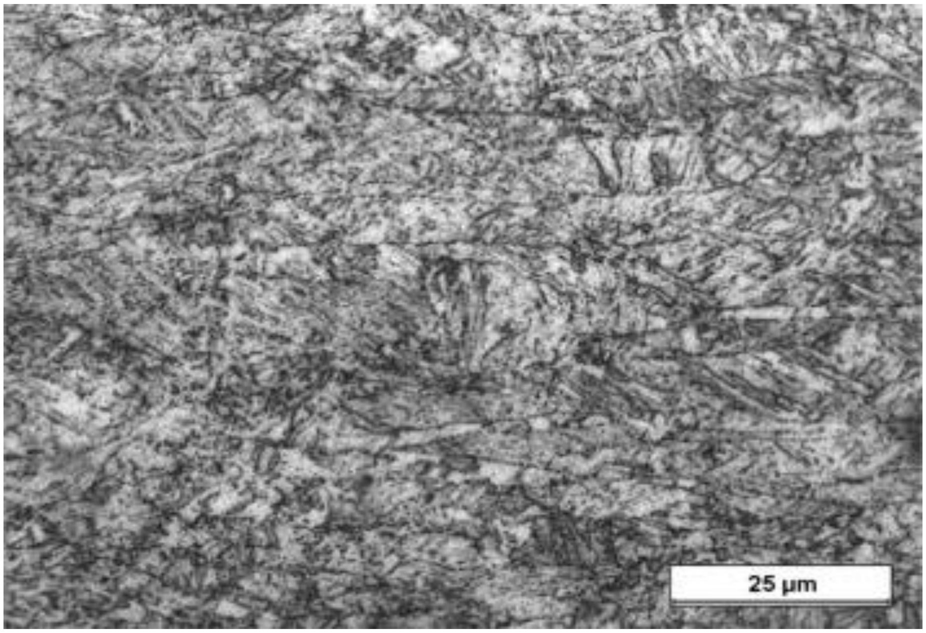
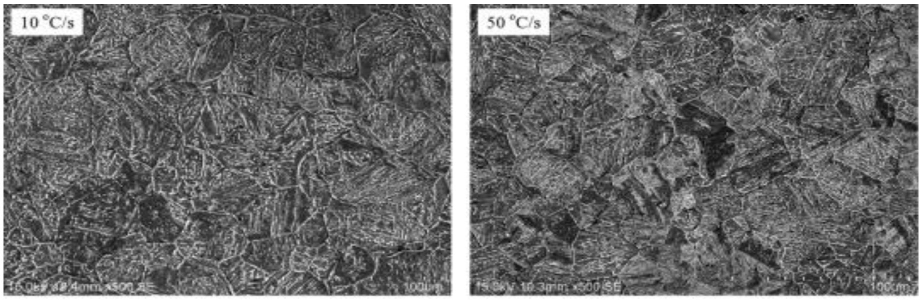


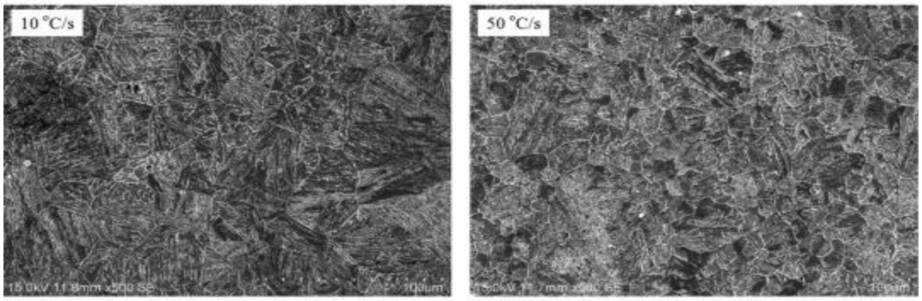
Figure 2.3: Microstructure of Q690 HSLA Steel

2.2.2. Effect of heating rate:

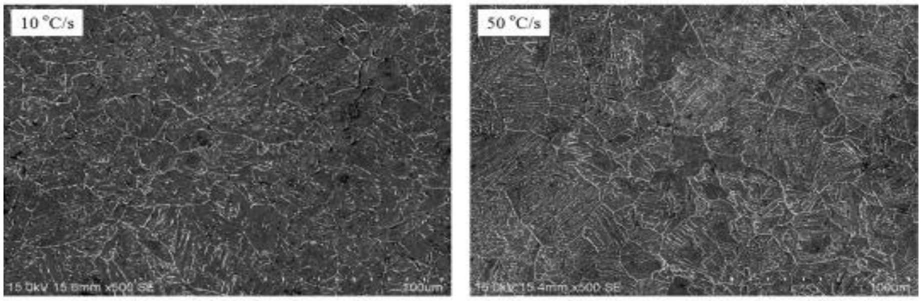
As mentioned above, heating rate also plays important role in high temperature because it also affects the microstructure of the end product. It is important in austenite homogenization and carbide dissolution, if the heating rate is very fast then the austenite don’t properly homogenize and it affect in the grain size and phase transformation. [3]



(a) ε=0



(b) ε=10%



(c) ε=40%

Figure 2.4: Microstructure of Q690 at Different Heating Rate and Deformation Rate.

Now we will take the example, in which sample Q690 is heated at different temperature rate i.e. 10 °C/s and 50 °C/s. From this we can see that at same deformation rate but at higher heating rate, there is less uniformity of grain sizes. There is both fine and coarse grains present in the microstructure when the heating rate is kept at 50 °C/s. However, same microstructure is obtained when there is no deformation but different heating rate. [3]

Fig. 2.4 (a) this figure shows the microstructure is granular and lath bainites both are present.

Fig. 2.4 (b) shows the microstructure of the sample with 10% deformation, we can see that polygonal ferrites are formed which are in small size and there is also some fine grained part is present when 50 °C/s heating rate is applied. Similarly, there is lath shaped microstructure formed when 10 °C/s heating rate is applied and some of the grains also in the form of oblong shape.

Fig. 2.4 (c) shows the deformation rate applied is 40% and it can be seen that there is a difference between microstructure when we vary the heating rate. There is improvement in the microstructure uniformity when the heating rate and due to this granular bainites microstructure is formed. Similarly, uneven grain sizes are obtained when uneven heating rates are applied and the microstructure formed are banded microstructure with number of ferrite block.

2.2.3. Decarburization principle and Depth profile:

Decarburization occurs in steel when the surface of the steel interacts with the atmosphere and carbon atoms at the surface react with the furnace atmosphere and removed in the form of gas from the steel surface. Carbon form the inner layer diffuses towards the surface because at surface the concentration of carbon is low, this process continues until maximum depth of decarburization has achieved. As the diffusion rate increases with temperature when the structure is fully austenitic, Maximum affected depth also increases as temperature rises above the Ac3. For temperatures in the two-phase region, between the Ac1 and Ac3, the process is more complex. As carbon diffuse differently in austenite and ferrite therefore decarburization is influenced by both composition and temperature. [4]

Decarburization decreases wear resistance and cause low fatigue property. To understand the depth of the decarburization, we should know about two characteristics: Free-ferrite layer depth (FFD) and partial decarburization depth (PDD). The sum of these two depths is called maximum affected depth (MAD). [4]

As mentioned above, this process depends upon temperature, composition and time. More the temperature more will be the diffusion of carbon from core to surface and more decarburization will occur. Usually in steels, **decarburization starts at 700 °C** and it increases with increase in temperature. Similarly if there are other alloying elements which are carbide formers then carbon will form carbide with them and decarburization is slowed and vice versa. In case of time, greater the time for steel at high temperature greater will be the chances for decarburization. As carbon will have time to diffuse towards surface. This dependency of decarburization can be explained with the help of example as shown in figure. [4]

In this example, a sample is heated at 700 °C for 2 hours and we see that there is negligible decarburization i.e. 50µm on the surface as there no free ferrite formation can be seen in optical microscope.

In second and third example, the sample is heated at 800 °C for 12 hours. In second example there is decarburization can be seen and free ferrite is up to 130 µm and in third there is more than 200 µm depth of decarburization can be seen. From this example it is clear that with increasing temperature and time the decarburization also increases.

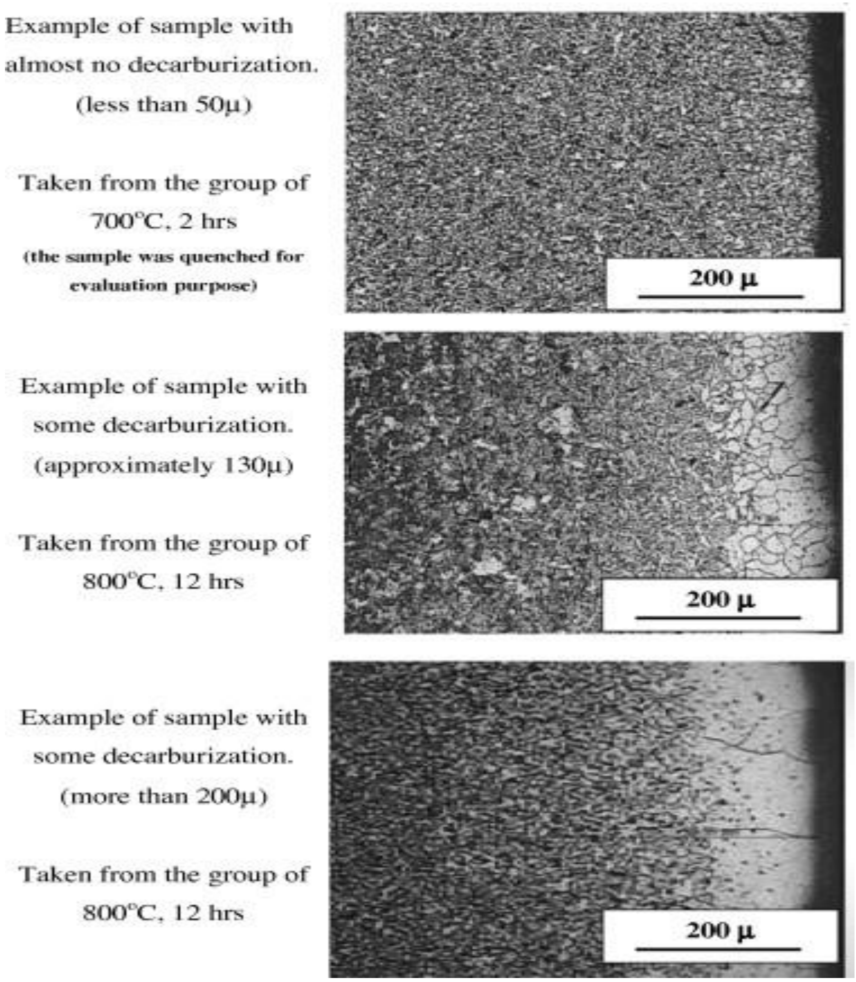


Figure 2.5: Microstructure of Steel Showing Decarburization at Different Temprature and Time

Decarburized surfaces of 5160 Mod austenitized at 1600°F for 80 min. and oil quenched. Free ferrite on the scale covered specimen, top. No free ferrite present on the specimen that was descaled before being austenitized bottom. [4] The reason is that, scale on the surface promotes oxidation of the carbon at the surface as oxygen is already present at the surface because of iron oxide and other alloying element oxide scale. So this is clear from the microstructure that scaled sample will show more decarburization rate as compare to descale.

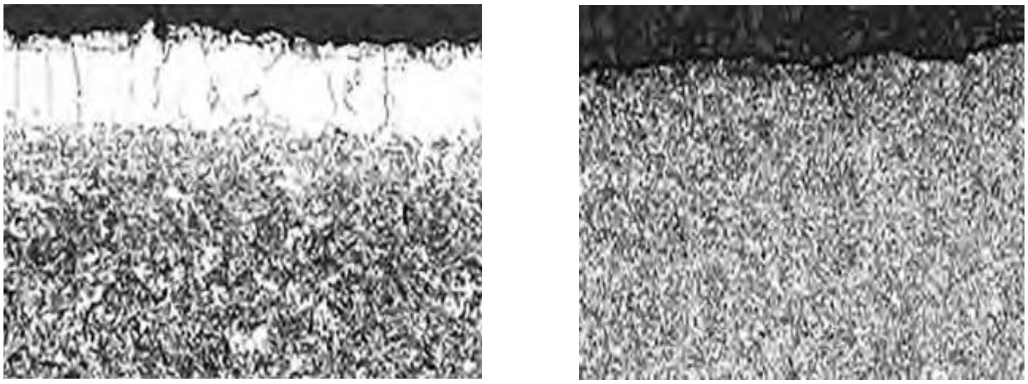


Figure 2.6: Scaled Covered Specimen on Left and Descaled Specimen on Right

2.3. Aluminum Alloys:

Aluminum is one of the most common and abundant element in the earth’s crust, more specifically metallic element. It nearly forms 8% of the entire earth crust. The aluminum most commonly present in nature in the form of bauxite or other compound which is then extracted from the ore after some processes. [5]

Aluminum is one the most important metallic element as its alloys are commonly used for structural application because of its high strength to weight ratio, good corrosion resistance, thermal and electrical conductivity.

Aluminums two most common alloys are 6061 and 6063, these alloys are usually have major percentage composition of magnesium and silicon after aluminum. Their most important properties are that they are hardenable, it means that we can make aluminum hard and strong by heat treatment process, which is carried out by precipitation hardening. [5]

Besides hardening mechanism, 6000 series alloys are artificially age hardened. In this, the material is first heated to form super saturated solution which is then heated in furnace at some specific temprature to artificially age hardened by formation of precipitate. The phenomena during artificially age hardening is given below:

- Clusters of Si atoms and clusters of Mg atoms.
- Guinier-Preston (GP) zones.
- Intermediate precipitate ß″.
- Intermediate precipitate ß′.

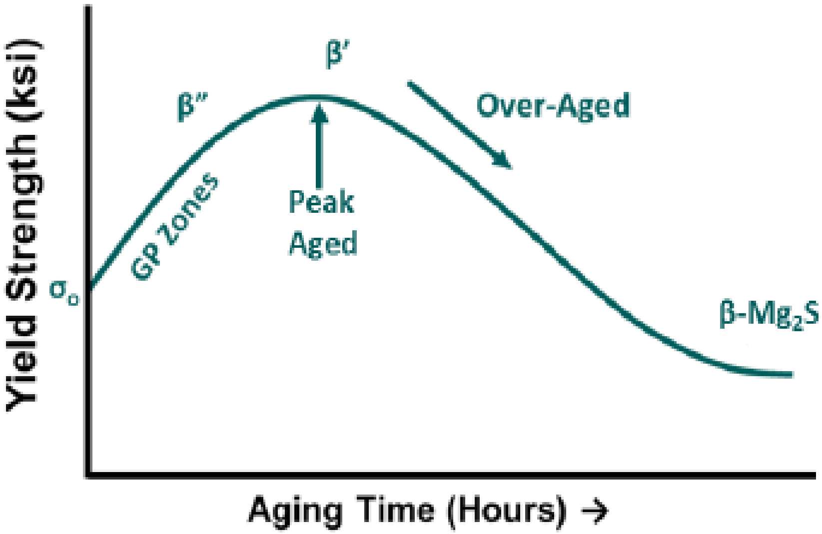


Figure 2.7: Yield Strength vs. Aging Time Relationship

The ß″ precipitates are coherent with the aluminum matrix, the ß′ precipitates are semi-coherent, and the ß-Mg2Si are incoherent. The strengthening mechanism is based on how easily a dislocation can move through the material. Any precipitate that impedes a dislocation from moving through the aluminum matrix will add strength to the alloy. [5]

When the precipitates are small and coherent with the aluminum matrix, dislocations shear through the precipitate. As the incoherency of the precipitates increases due to elevated temperature and time, the dislocations have to bow around the precipitate. [5]

The top of the peak is referred to as the peak age condition, and is where the maximum strength of the material is found. Past this point where the equilibrium phase is reached is known as over-aging. Before the peak where the ß″ phase is present is known as under-aging. [5]

Over-aging results in a loss of strength because as the precipitates grow larger, the overall amount of precipitates in the system lowers. The particles are too large to be sheared, so they are by passed by dislocations moving through the material. [5]

Thus this phenomena shows that how the physical properties can be varied by temperature applications. [5]

2.4. Graphite:

As mentioned above, there is very less work and data present on graphite which can be used as structural application. Graphite has applications for high temperature just like rocket nose, Moderators in nuclear reactors but these are not structural applications. For load bearing structural material at high temperature, very few work has been done, if it has done it is confidential.

Graphite is very light weight as compare to metals due to its very low density. Due to this reason it can give high specific strength at high temperatures where steel and aluminum strength start to decrease. Graphite show various mechanical and physical properties. It has moderate strength at normal temperature but it shows anomalous behavior and its strength increases with increase in temperature instead of decreasing and after some specific temperature it starts to decrease. [6]

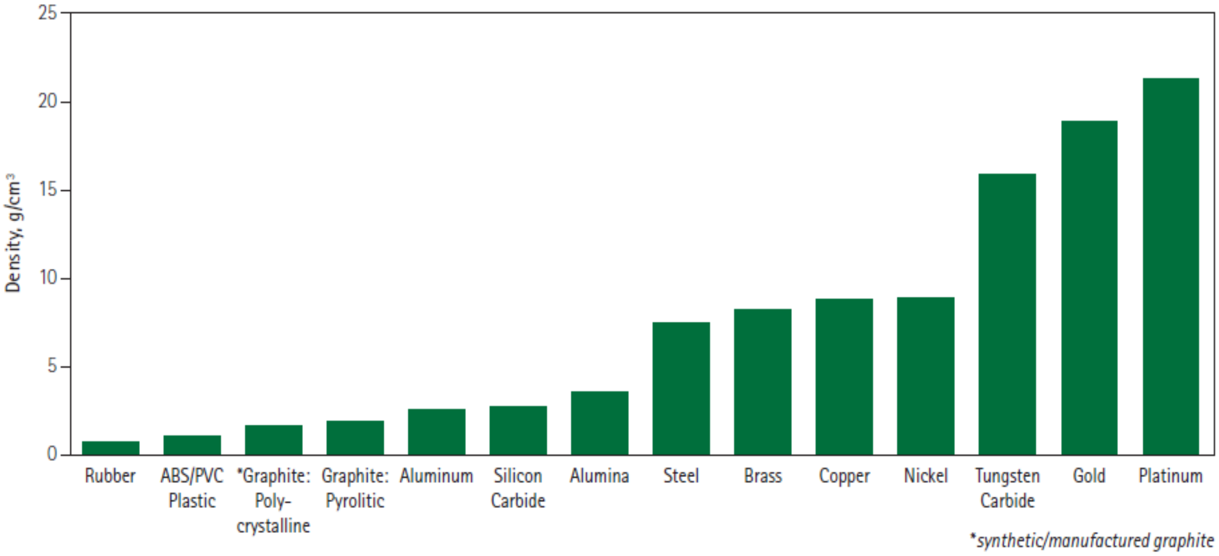


Figure 2.8: Density Variation of Different Engineering Materials

2.4.1. Temperature Effect:

In metals, the tensile strength decreases with increase in temperature, this is the natural behavior of the metals while it is different in case of graphite. The tensile strength of graphite increases instead of decreasing as in the case of metals. This behavior of graphite is due to its characteristic and it is not same for each type of graphite. Some graphite have larger effect of temperature while some graphite has very low effect.

In an inert atmosphere, AXF-5Q shows graphite shows tensile strength of approx. 137 MPa at 2370°C while it show tensile strength of 63 MPa at room temperature. From this we can see that with increase in temperature the strength of the graphite also increases. This behavior can be seen in the figure

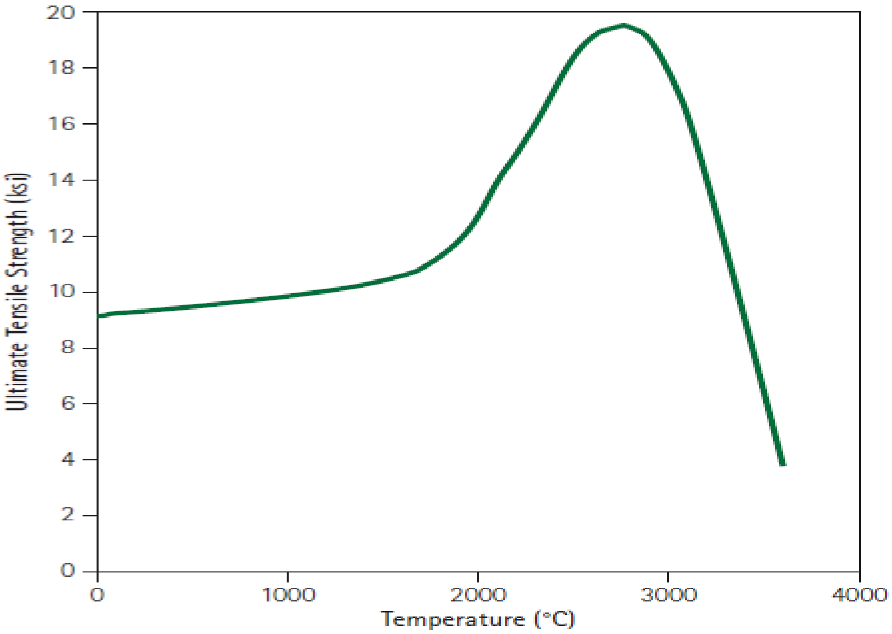


Figure 2.9: Effect of Temperature on Ultimate Tensile Strength of AXF-5Q Graphite.

2.4.2. Carbon-Carbon composite as structural material:

Carbon-carbon composite shows structural integrity at higher temperatures. As compare to conventional graphite mechanical properties of C-C composite is much superior. Three dimensional Carbon-Carbon composite can with stand damage and under interlaminar shearing less crack growth occur. Its tensile strength increases at high temperature i.e. above 1200°C where other metals or super alloys fails. While this material don't fails abruptly or catastrophically just in case of metals or super alloys, it goes through gradual failure. [6]

Heat treatment also affects the mechanical properties of the C-C composite and graphite. When we carbonize the material at 1000°C and then graphitization at 2700°C, there is an increase in flexural strength up to 54%, decrease in interlaminar shear strength up to 40% and 93% flexural modulus increase. The tensile and flexural properties of depends upon the orientation and dimension of the fibers of the C-C Composite. [6] [7]

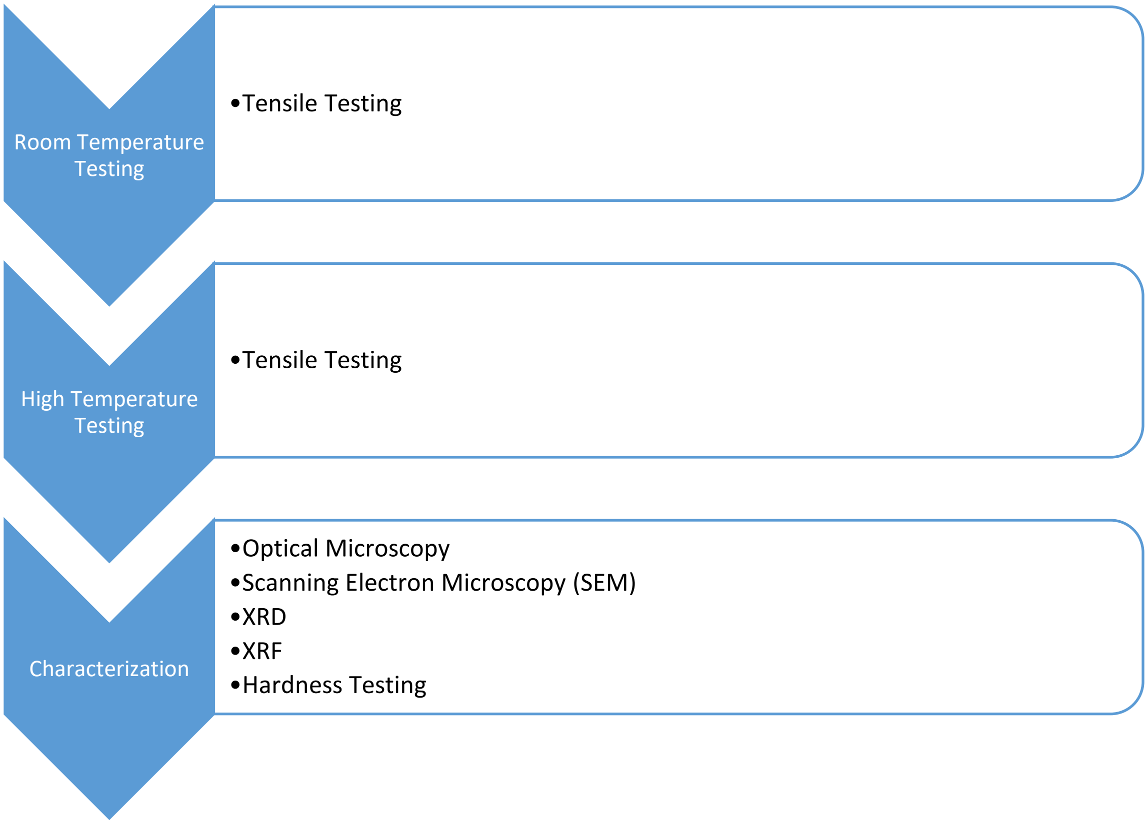
2.4.3. C-C composite oxidation:

There is only one drawback about C-C composite at high temperature. This drawback is its susceptibility to oxidize at high temperature, the oxidation starts to occur above 500°C which gradually increases and becomes sever with increase in temperature up to 800°C. The rate of oxidation is limited only by the diffusion rate of oxygen through the surrounding gas to the carbon surface. In applications like rocket motors this is not very important; however, in space plane and as turbine engine material the development of extended lifetimes of around 100 h is required. [6]

CHAPTER 3

EXPERIMENTATION

In order to carry out experiment, three different materials Aluminum alloy (Al 6061), HSLA Steel (ASTM 4240 Steel) and Graphite (C-C Composite) were taken. The experiments were performed in two phases. In first phase, it was performed at room temperature, in which tensile testing and characterization of the materials were performed so that verification of the material could be done in relation with literature and also condition of the material can be confirmed in as received condition. In second stage, the materials were tested at high temperature and then characterization were performed. The following flow chart demonstrates the testing and characterization steps which was followed throughout the project:



3.1. Room Temperature Testing:

In room temperature tensile testing, the materials were cut according to the ASTM E8M standard on the CNC machine. Three samples of each materials were cut and total nine samples were prepared so that average best value of the results could be taken and error could be minimized. Tensile testing was performed on the 300 KN UTM machine (Tinius Olsen super 602L). In order to avoid the overheating of the machine the strain rate of 3 mm/s was set.

Figure 3.1 Tensile Testing Machine

3.2. High Temperature Testing:

In order to find out the high temperature mechanical properties of these material, high temperature tensile testing was performed. This was carried out on high temperature tensile testing machine (50 KN SHAMIDZU AO-I) which was specially built for this purpose. Nine samples of each material (HSLA Steel, Aluminum Alloy and Graphite) and total twenty seven samples were prepared by following the standard ASTM E21. These samples were then tested on different temperature ranges i.e. 100 °C, 200 °C and 300 °C. The strain rate of 1 mm/s was set for the testing. The results then recorded which are obtained from the testing. [7]

Figure 3.2 High Temperature Tensile Testing Machine.

3.3. Characterization:

Characterization was performed in order to study the materials structure and their properties. It was performed in both stages, i.e. at room temperature and at high temperature. First at room temperature, it was carried out in order to verify the material properties and its structure in as received condition, so that it could be cross checked with the required properties.

Similarly, characterization was performed for the high temperature tested material so that high temperature behavior and changes could be studied in the materials.

Following Characterization was performed on the materials.

3.3.1. Optical Microscopy:

Optical microscopy was performed to study the microstructure of the materials. Each material was prepared and the checked under the light microscope. First each sample was grind on different grain size sand papers. The grain size number used was 120, 240, 400, 800, 1200. After grinding the materials, these materials were polished on the polisher by using 0.5 micron alumina powder until mirror look came. For each material, there was different etchant to visualize the grain boundaries and microstructure.

Figure 3.3 Optical Microscope

For HSLA Steel, 3% Nital solution was used which contain 3% Nitric acid and 97% Ethanol. The solution was placed on the polished sample for 10-15 seconds and it was washed and dried with hand dryer. After that it was checked under microscope.

Similarly for Aluminum 6061, etchant was made which contain 25ml Methanol, 25ml Nitric acid, 25ml Hydrochloric acid and 1 drop of Hydrofluoric acid. The solution was placed on the sample for 60 seconds and then washed and died with hand dryer.

3.3.2. Scanning Electron Microscopy:

In order to check the microstructure at high resolution, SEM was performed because microstructure was not clear on optical microscope. The prepared sample was placed in SEM machine and then microstructure was analyzed at high resolution.

3.3.3. XRD:

An analytical technique that is used to identify phases of crystalline material and provides the unit cell dimension or d-spacing is known as X-ray powder diffraction (XRD). The working principle behind XRD is that the crystalline substances diffract the incoming x-ray wavelengths in three dimensions similar to inter planner spacing in a crystal lattice. In XRD, monochromatic X-rays are used that are generated from a cathode ray tube in the direction of the sample. [7]

When the condition suffices the Bragg's law i.e. constructive interference takes place due to the interaction of the sample and the incident rays and the diffracted rays are then detected and processed. The scanning is done through a range of 2 theta angles. The diffraction peaks are converted to d-spacing which allows the identification of the sample because each mineral has a unique set of d-spacing. This is possible due to the comparison with the standard reference patterns. Generally, CuKα radiation = 1.5418Å are used for XRD. A graph of 2-theta and counts per second is plotted after XRD is done. This is how XRD works. [7]

Figure 3.4 XRD Machine

3.3.4. XRF:

XRF is a technique for elemental analysis, in which fluorescence is used. The basic working principle of the technique is that the when atoms of any element are given energy, they get excited. Under the influence of this excited state, there is an emission of X-ray photons which are characteristic to that element therefore allowing the identification of elements and their quantity. [8]

Similar to SEM an electron beam is used, which when strikes the surface produces photons and photoelectron. Theses photoelectrons are produced from the shells near to nucleus (K shells) as defined by Bohr model) and the gap between these shells and the outermost shell is compensated by their kinetic energy, the difference between the binding energy and the incident energy. [9]

Since an electron has left the atom a hole is produced, and to fill this space an electron migrates. This migration of the electron leads to the production of fluorescence.

The energy of the ray emitted is equal to the difference between the excited state energy and relaxed state energy of the electron i.e. the initial and the final state. Now this photoelectric absorption can also cause production of Auger electron, as that is another way the atom can move to the relaxed state. A spectroscopic data is formed to determine the probability of getting the photons produced instead, determined by the fluorescence yield. And that is on what the XRF machine is built [9]

3.3.5. Hardness Testing:

Hardness test was carried out to check the hardness value of the materials. As HSLA Steel and Aluminum alloy are metal so for these material Rockwell hardness testing was carried out. For HSLA steel, Rockwell C scale was used as it is a hard material while for Aluminum Rockwell B scale was used as it is a soft material. For C-C composite (Graphite), knoop test was carried out as it was a composite and Rockwell testing cannot be carried out for composite. In this case, across the fiber and along the fiber both hardness values was obtained.

CHAPTER 4

RESULTS AND DISCUSSION

4.1. Results:

The results which are obtained from the experimentation have been discussed in the section below and further comparison of results is discussed in the discussion section.

4.1.1. Room Temperature Testing:

4.1.1.1. Aluminum Alloy:

Series of experiments were performed on aluminum and results of those experiments are as follows:

4.1.1.1.1. Tensile Testing:

Tensile testing was performed on the three samples of the Aluminum 6061 samples and the results obtained were somehow close to those which were observed during the literature review. 6061-T6 tempered alloy of Aluminum gave tensile strength of 310 MPa followed by the Yield strength of 276 MPa. Overall elongation observed for this alloy was of 12% maximum. The results are shown in the table below (Table 4.1):

Table 4.1: Room Temperature Aluminum 6061 Tensile Testing Results

Sample No.	**Tensile Strength (MPa)**	**Yield Strength (MPa)**	**Percentage Elongation (%)**
1.	308	277	9
2.	311	264	13
3.	311	288	14

4.1.1.1.2. Hardness Testing:

Rockwell Hardness Testing Machine was used for the measurement of the Hardness value of the 6061-T6 Aluminum alloy with the B scale giving the final value of **HRB 55**. The values for hardness are given in the table below (Table 4.2):

Table 4.2: Hardness Values

No. of Sample	Hardness Value (HRB)
1.	56.5
2.	54
3.	54.5

4.1.1.1.3. XRF Analysis

Through XRF we found the major precipitate forming elements and they were Si, Mg, Fe and Cu respectively. The elemental composition of the Aluminum 6061 have been shown in the Table 4.3 below:

Table 4.3: XRF Table for Aluminum 6061

Element	Al	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti
Composition	**97.05**	**0.63**	**0.4**	**0.32**	**0.06**	**0.97**	**0.28**	**0.13**	**0.12**

4.1.1.1.4. XRD Analysis

This analysis was done to identify the phases of the crystal structures which were forming during the various temperature ranges. Peaks of Al were observed and were later read to know the possible phases present. From the XRD pattern it is came to know that the major phases present are of Aluminum, Mg_2Si and FeMnSiAl. The XRD graph of Aluminum 6061 have shown in figure 4.1 below:

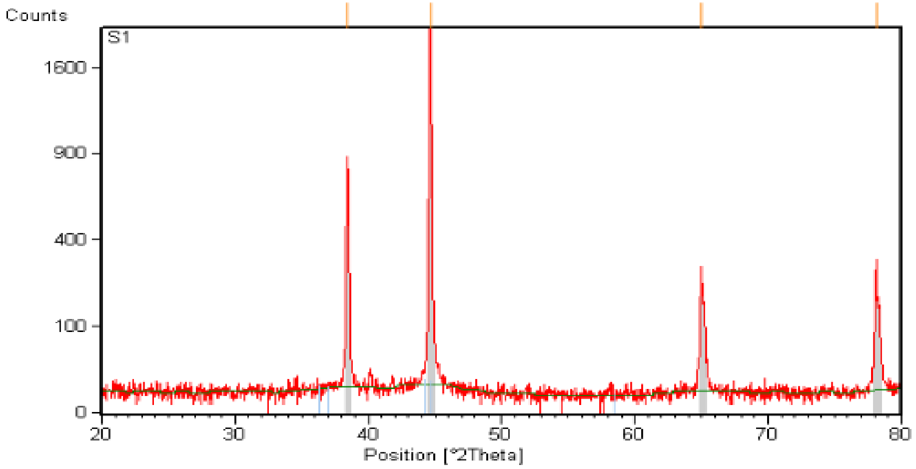


Figure 4.1: XRD Graph of Aluminum 6061

4.1.1.1.5. Microstructure Analysis:

At first microstructure was observed through the optical microscopy which shows the grain boundaries present in abundance and along them precipitates are also present which are ready to disperse in the matrix of the aluminum. As it can be seen in the optical microscope graph as shown in figure 4.2:

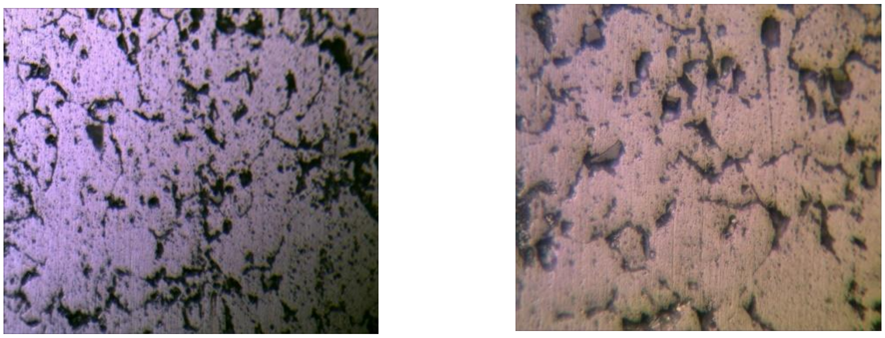


Figure 4.2: Microstructure of Aluminum 6061 500X on left and 800X on right

SEM analysis was done to know about the precipitates formation at room temperature at high reolution and also identification of them along the grain boundaries at first and later dispersion. The precipitates were in the globular form due to one of the major alloying element i.e. copper. The reason for globular structure is that the Aluminum alloy is T6 tempered, from this globular struture it is confirmed.

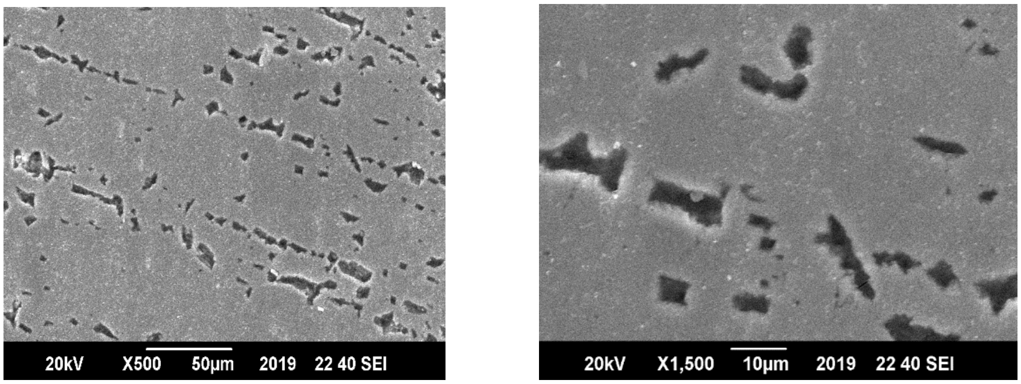


Figure 4.3: SEM of Aluminum 6061 500X on left and 1500X on right.

4.1.1.2.HSLA Steel:

4.1.1.2.1. Tensile Testing:

Ultimate Tensile Testing machine was utilized to obtain values for the Yield strength and the Tensile strength and it was observed to reach on average value of 1400 MPa and 1307 MPa respectively with maximum elongation of 12%. The results shows that the HSLA Steel is quenched tempered. The room temperature results have been shown in the table 4.4 shown below.

Table 4.4: Room Temperature HSLA Steel Tensile Testing Result

Sample No.	Tensile Strength (MPa)	Yield Strength (MPa)	Percentage Elongation (%)
1.	1413	1320	10
2.	1402	1307	12
3.	1390	1300	11

4.1.1.2.2. Hardness Testing:

Rockwell Hardness Testing was performed for the ASTM 4240 HSLA steel on the C scale and this gave us the hardness value of HRC 39 on average for the three samples that we tested for this experiment. The hardness values are given in the table 4.5 below:

Table 4.5: HSLA Steel Hardness test results

No. of Sample	Hardness Value (HRC)
1.	35
2.	43
3.	39

4.1.1.2.3. XRF Analysis:

XRF analysis was performed in order to verify the elemental composition of the HSLA Steel. Through XRF we found the major alloying elements in the steel and their composition. The elemental composition of the HSLA Steel have been shown in the table 4.6 below:

Table 4.6: XRF Table for HSLA Steel

Element	Fe	C	Mn	Si	Cr	Mo	S	P	V	Cu	Ni
Composition	**95.47**	**0.29**	**0.85**	**1.5**	**1.12**	**0.5**	**0.009**	**-**	**0.08**	**0.16**	**0.02**

4.1.1.2.4. XRD Analysis

X-Ray Diffraction of the steel samples was done to identify all the possible phases present inside. Two major peaks were obtained and later comparison was done with the base peak of the obtained results. When the graph is analyzed in the software it is confirmed by the software that the major phase present in the material is BCT which is martensite. The largest peak in the graph is of martensite and it is confirmed from the XRD result that the material in as received condition is quenched tempered due to the presence of martensite (BCT) as major phase. The XRD graph is given Below in Figure 4.4:

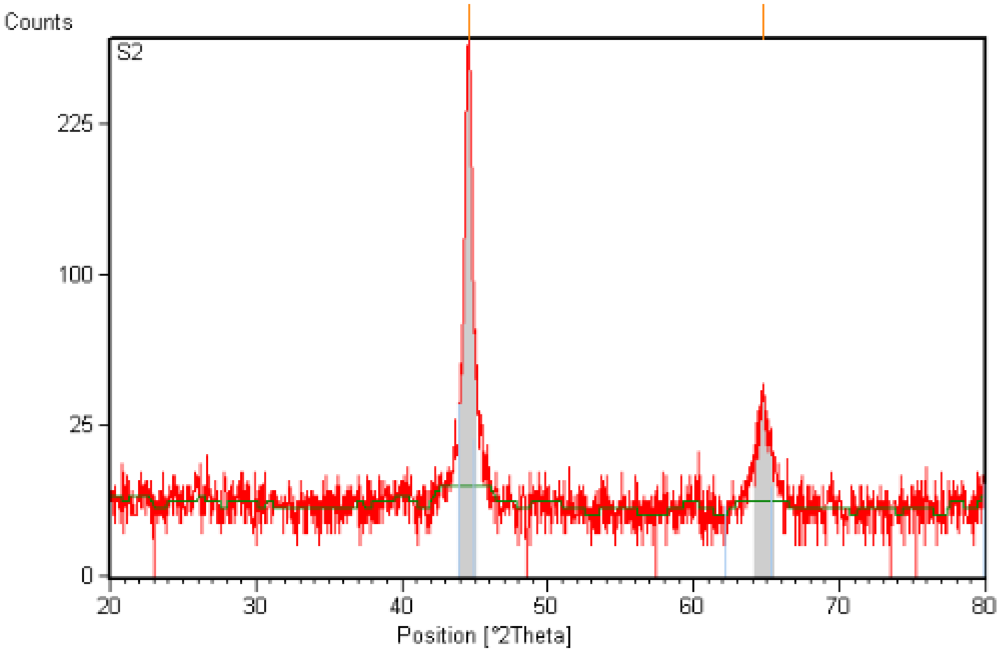


Figure 4.4: XRD Graph of HSLA Steel

4.1.1.2.5. Microstructure Analysis

At first etched samples in the as-received conditions were observed in the optical microscopy to know more about the microstructure of the material. As from the XRD pattern we have seen that the major phase present is of martensite so in order to confirm this microstructure analysis was done. In Optical microscopy, we see that there is sharp needle like microstructure were present which was lathe structure from this we confirmed that the material in as received condition is quenched tempered. And also there were also sign of decarburization present on the surface of the material. When microstructure was analyzed there was free ferrite present on the surface as their microstructure can be seen as bright region. So, decarburization also confirmed from the microstructure analysis.

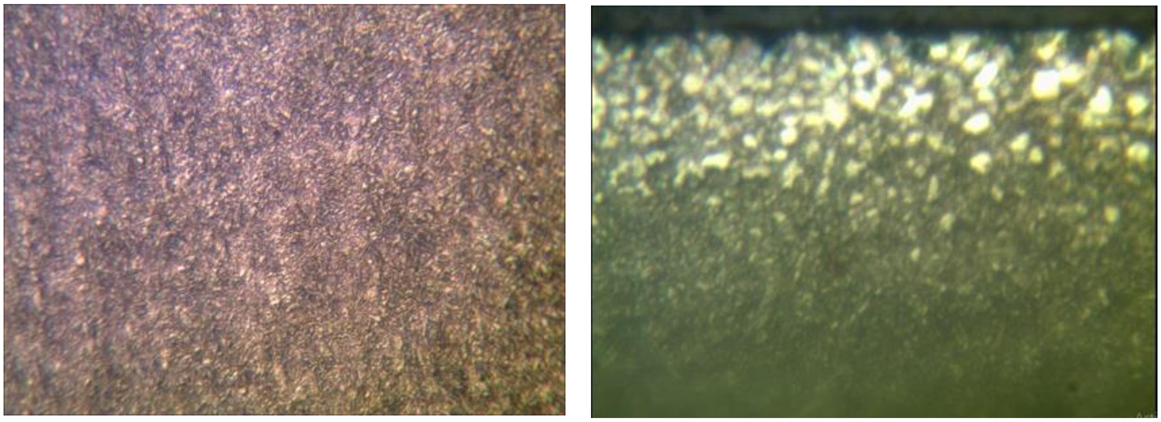


Figure 4.5: Microstructure of HSLA 500X left and 800X (Side view right)

Further analysis was also done through the SEM images in high resolution of the as received HSLA steel samples with varying magnification powers. Which confirmed the martensite phase and quenched tempered state of the material.

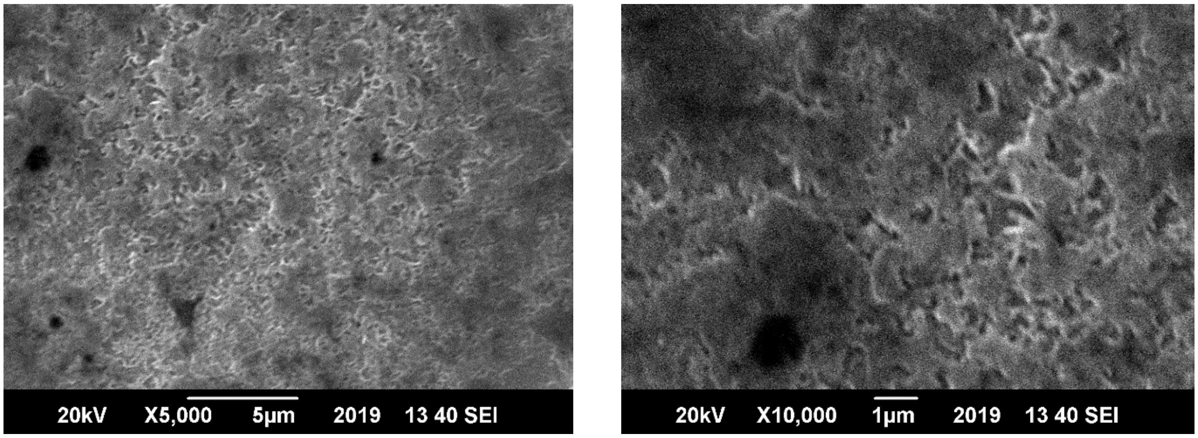


Figure 4.6: SEM of HSLA steel 5000X on left and 10000X on right

4.1.1.3. Graphite (C-C Composite):

4.1.1.3.1. Tensile Testing:

At room temperature testing, the tensile strength of graphite was not comparable with HSLA steel as it has very low strength as compare to steel. But its anomalous behavior is the reason we are comparing it with steel and aluminum so the room temperature study is to verify the composite tensile strength with the literature. Further high temperature testing will explain why we chose graphite for high temperature application.

Table 4.7: Room temperature graphite tensile testing result.

Sample No.	Tensile Strength (MPa)	Yield Strength (MPa)	Percentage Elongation (%)
1.	623	568	5
2.	605	543	2
3.	602	539	2

4.1.1.3.2. Hardness Testing:

Knoop Hardness Testing machine was used and hardness values were observed in two different directions across the fibre and along the fibre. Knoop testing was performed as Rockwell testing is for metals and cannot be performed on composite therefore knoop test was performed. We observed maximum hardness to be in the direction where fibres were aligned in the perpendicular direction. The values are given in Table 4.8

Table 4.8: Graphite hardness results

No. of Sample	Hardness Value (GPa) [Across]	Hardness Value (GPa) [Along]
1.	0.73	0.24
2.	0.72	0.23
3.	0.59	0.19

4.1.1.3.3. XRD Analysis:

This analysis was done to identify the phases of the crystal structures which were forming during the various temperature ranges. One major peak was observed and was later read to know the possible phases present. The pattern is shown in figure 4.7

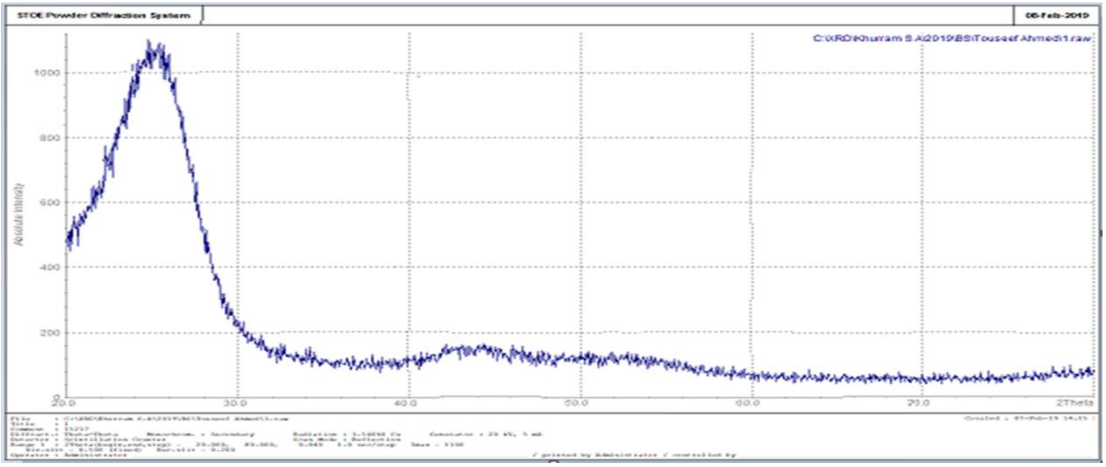


Figure 4.7: Graphite XRD graph

4.1.1.3.4. Microstructure Analysis

SEM analysis of the C-C composite showed the alignment of the fibers in the matrix of the composite. Magnification power was increased further to have their clear observation.

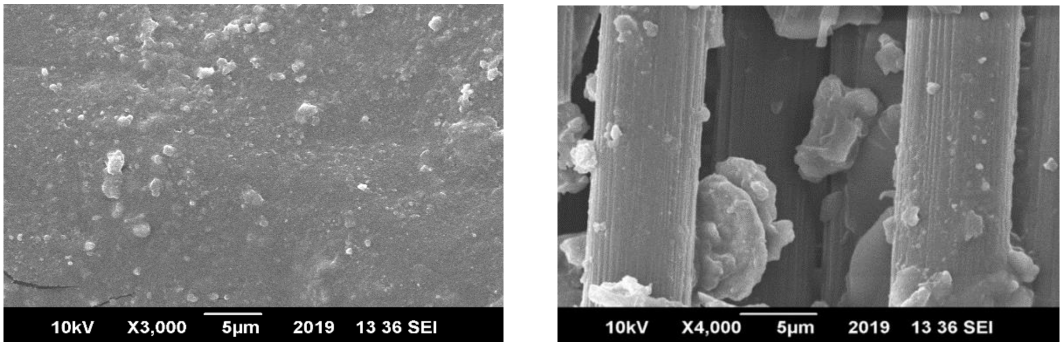


Figure 4.8: SEM of Graphite, 3000X on left and 4000X on right

4.1.2. High Temperature Testing:

4.1.2.1.Aluminum:

4.1.2.1.1. High Temperature Tensile Testing:

For the 6061-T6 tempered series of the Aluminum alloys it was observed that with increasing temperature we are observing reduced mechanical strength. At room temprature it has Tensile strength of 310 MPa and Yield strength of 276 MPa and with increase in temperature both yield strength and tensile strength reduced to 55 MPa and 69 MPa till 300°C with maximum elongation of 40%. As mentioned before, for each temperature range 3 samples were prepared their results are shown in the table 4.9, 4.10 and 4.11 given below:

At 100 **°C:**

Table 4.9: High Temperature testing of Aluminum 6061 at 100 °C

Sample No	Tensile Strength (MPa)	Yield Strength (MPa)	Elongation (%)
1	230	185	14
2	223	180	13
3	256	198	15

At 200 **°C:**

Table 4.10: High Temperature testing of Aluminum 6061 at 200 °C

Sample No	Tensile Strength (MPa)	Yield Strength (MPa)	Elongation (%)
1	160	110	21
2	168	115	22
3	162	111	20

At 300 **°C:**

Table 4.11: High Temperature testing of Aluminum 6061 at 300 °C

Sample No	**Tensile Strength (MPa)**	**Yield Strength (MPa)**	**Elongation (%)**
1	69	55	40
2	60	50	38
3	68	55	40

After getting results at different temperature, the average or most suitable values have been selected which have been shown in the table 4.12 below:

Table 4.12: High Temperature Tensile Testing of Aluminum at Different Temperature Ranges

Temperature (°C)	**Tensile Strength (MPa)**	**Yield Strength (MPa)**	**Elongation (%)**
25	310	276	12
100	230	185	14
200	168	110	20
300	69	55	40

4.1.2.2. HSLA Steel:

4.1.2.2.1. Tensile Testing:

HSLA steel also showed same behavior as that of 6061-T6 tempered Aluminum alloy.

Maximum elongation is of 25% and we observe that yield strength decreased to 190 MPa from 1307 MPa with increasing temperature from 25°C to 300°C. The results for different samples at different temperature ranges are given below in table 4.13, 4.14 and 4.15:

At 100 **°C:**

Table 4.13: High Temperature testing of HSLA Steel at 100 °C

Sample No	Tensile Strength (MPa)	Yield Strength (MPa)	Elongation (%)
1	900	740	14
2	895	730	15
3	907	742	15

At 200 **°C:**

Table 4.14: High Temperature testing of HSLA Steel at 200 °C

Sample No	Tensile Strength (MPa)	Yield Strength (MPa)	Elongation (%)
1	400	270	18
2	402	270	19
3	414	280	18

At 300 **°C:**

Table 4.15: High Temperature testing of HSLA Steel at 300 °C

Sample No	Tensile Strength (MPa)	Yield Strength (MPa)	Elongation (%)
1	290	190	25
2	292	187	23
3	288	186	24

After getting results at different temperature, the average or most suitable values have been selected which have been shown in the table 4.16 below:

Table 4.16: High Temperature Tensile Testing of HSLA Steel at Different Temperature Ranges

Temperature (°C)	Tensile Strength (MPa)	Yield Strength (MPa)	Elongation (%)
25	1400	1307	14
100	900	740	15
200	400	270	18
300	290	190	25

4.1.2.2.2. Microstructure Analysis:

Through SEM microstructure of the samples was observed to verify the formation of ferrites from martensite as with increase in temperature the martensite start to transform into ferrite because the epsilon carbon start to diffuse outside from the martensite due to which the martensite transforms into ferrite and losses its tetragonality and the strength of the material start to decrease.

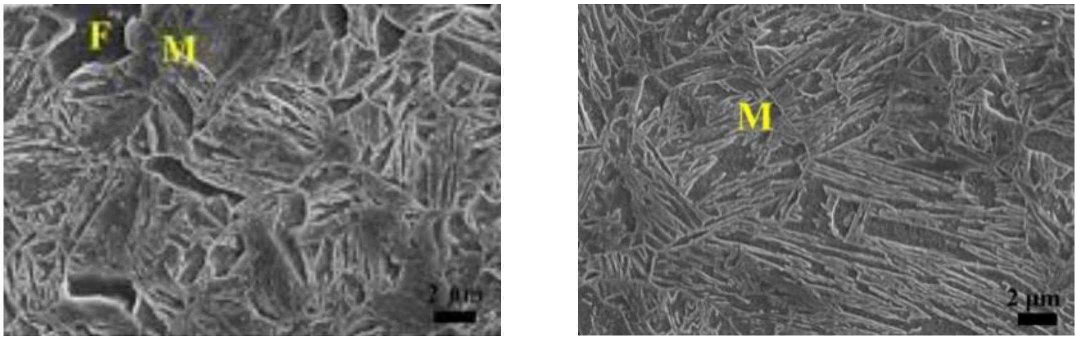


Figure 4.9: SEM of HSLA Steel showing Martensite (M) and Ferrite (F)

This demonstrates the needle like structure of the martensite which are responsible for the strength and also showing the ferrite region which are inducing softness and decrease in strength. [3]

4.1.2.3. Graphite (C-C Composite):

4.1.2.3.1. Tensile Testing:

During the high temperature testing of the C-C Composite for the high temperature testing we observed the mechanical strength was increasing anomalously. With every 100°C we had 300 MPa increase in the tensile strength and the yield strength approximately. Maximum elongation which was observed was of 8%. The results for different samples at different temperature ranges are given below in table 4.17, 4.18 and 4.19.

At 100 **°C:**

Table 4.17: High Temperature testing of Graphite at 100 °C

Sample No	Tensile Strength (MPa)	Yield Strength (MPa)	Elongation (%)
1	900	708	5
2	905	709	5
3	896	700	5

At 200 **°C:**

Table 4.18: High Temperature testing of HSLA Steel at 200 °C

Sample No	**Tensile Strength (MPa)**	**Yield Strength (MPa)**	**Elongation (%)**
1	1300	1020	6
2	1304	1010	6
3	1315	1025	7

At 300 **°C:**

Table 4.19: High Temperature testing of Graphite at 300 °C

Sample No	**Tensile Strength (MPa)**	**Yield Strength (MPa)**	**Elongation (%)**
1	1600	1470	8
2	1590	1450	7
3	1612	1480	8

After getting results at different temperature, the average or most suitable values have been selected which have been shown in the table 4.20 below:

Table 4.20: High Temperature Tensile Testing of Graphite at Different Temperature Ranges

Temperature (°C)	**Tensile Strength (MPa)**	**Yield Strength (MPa)**	**Elongation (%)**
25	610	550	3
100	900	708	5
200	1300	1020	6
300	1600	1470	8

This data was later plotted on the graph to observe the behaviour of all the samples.

4.2. DISCUSSION:

Aluminum 6061-T6 tempered series and the HSLA Steel which are readily being used in the structural applications shows drastic decrease in the strength with the increasing temperature. But on the other hand the C-C composite shows anomalous behavior of increase in mechanical strength with rising temperature. This was further studies by performing experimentations on the samples in the temperature range of 100°C to 300°C. This behavior of the C-C composite is important to study as major applications are dependent on this property.

4.2.1. Aluminum:

The data which was obtained through the tensile testing was utilized to plot a stress-strain graph in Figure 4.10. The line graph clearly depicts the maximum tensile strength of 310 MPa following the strain rate of 3mm/sec. The tensile strength keeps on increasing with the load application till it reaches the fracture point.

This behavior of alloys of Aluminum is because of the formation of the precipitates which hinders the dislocation movement in the crystal structure. The matrix of Aluminum alloys is of FCC structure while the precipitates are of BCC. These precipitates are dispersed in the matrix starting from the grain boundaries. [5]

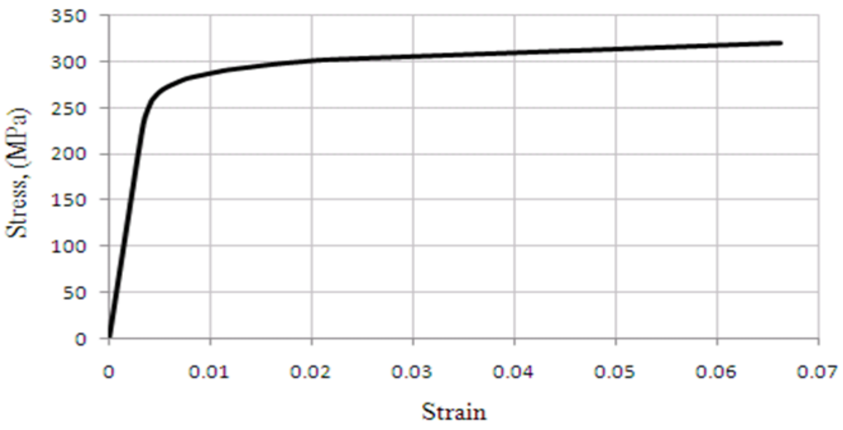


Figure 4.10: Stress strain Graph of Aluminum 6061 at Room Temperature.

But when temperature was increased from 50°C to 300°C this behavior was not the same as shown in figure 4.11 that of the newly formed precipitates started leaving the grain boundaries and settled on the matrix by dispersing themselves. With further rise in temperature grain coarsening started and thus the Aluminum alloy lost major strengthening factors in its structure. This can be explained through the figure 4.11 below.

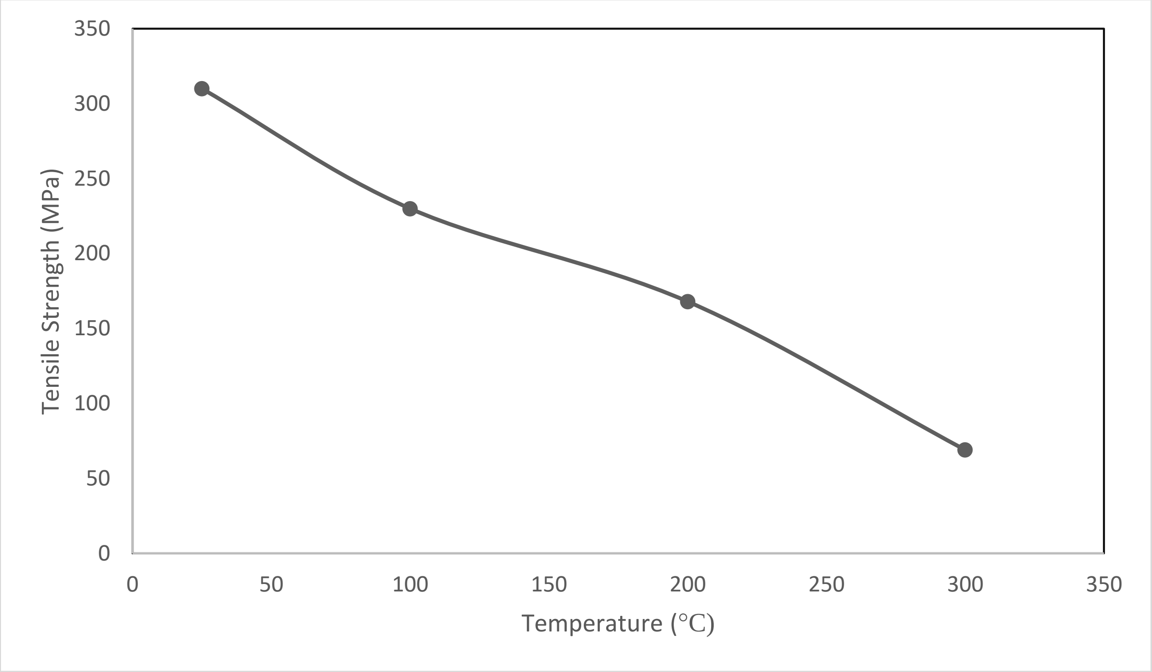


Figure 4.11: Temperature vs. Strength Plot of Aluminum 6061

4.2.2. HSLA Steel:

Studying the microstructure of the as received samples of the HSLA steel samples we observed that the microstructure is of lathe martensitic further showing sharpness in it. This was also confirmed by the XRD analysis as we observed the BCT structure and the hardness value of HRC 60 was also indication for the material to be quenched tempered. [10]

But when we tested same sample at high temperature we observe decrease in mechanical strength. This is because as of diffusion which makes the structure unstable as carbon starts moving outside from the BCT structure and thus tetragonality is reduced. All of this epsilon carbon gets removed and structure transforms from BCT to BCC. [2]

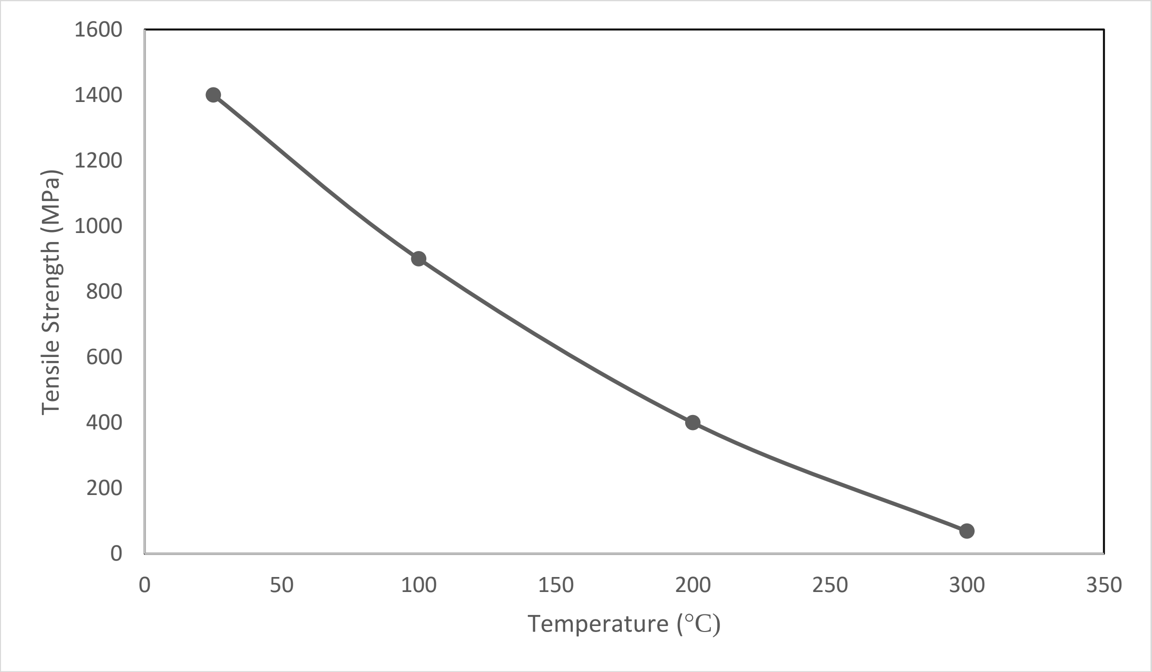


Figure 4.12: Temperature vs. Strength Plot of HSLA Steel

Decarburization also adds on the decrease in the mechanical strength as carbon gets oxidize leaving free ferrite behind which is a softer phase. This is the reason why we have drastic decrease in the mechanical strength from 1400 MPa to 210 MPa in the temperature range of 50°C-300°C.

4.2.3. C-C Composite:

These composites have hexagonal structure having covalent bond which is fixed with the other atoms in a parallel manner through the van der wall forces. When temperature is increase we observe that these van der wall forces start weakening and the layers of the structure kept sliding further decreasing the density along with the oxidation phenomenon which is also happening at the same time. [10]

C-C Composite possess 2D and 3D structure which gives strain hardening effect at room temperature but when we go to higher temperature we observe weakening of the layer which are bonded together would cause in strain hardening effect by interlocking with each other. This results into the decrease in the porosity which is further responsible for the increase the strength. This was the anomalous behavior due to which the strength of the graphite increases instead of decreasing with increase in temperature. [10] The trend of strength of graphite in relation with temperature is shown in the figure 4.13 given below:

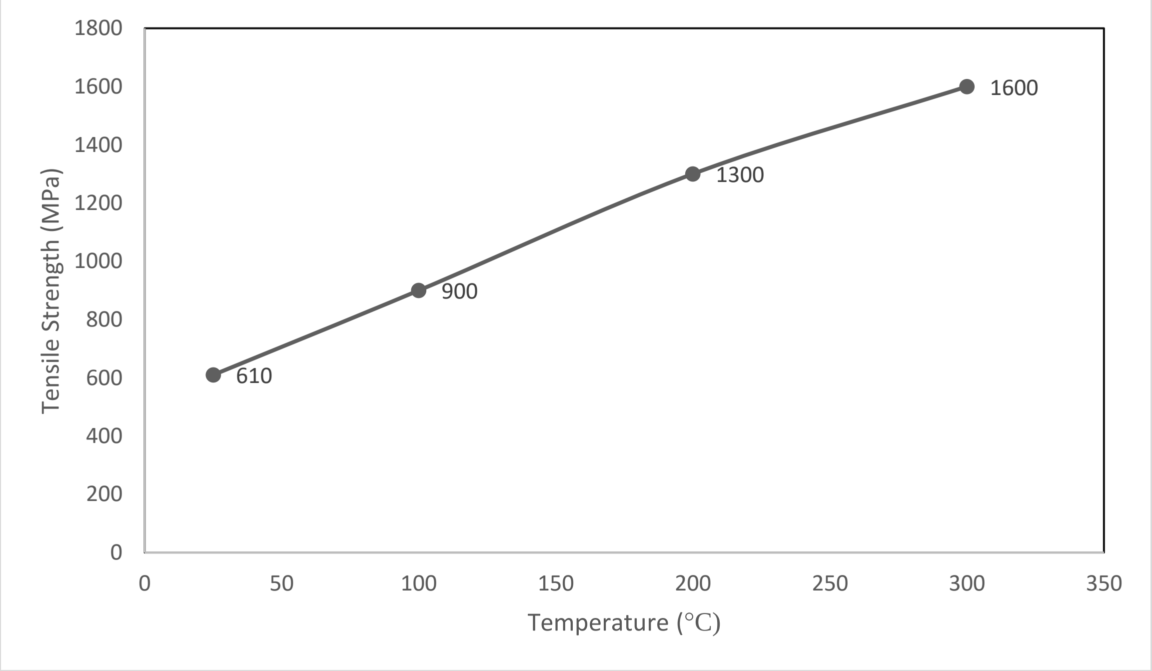


Figure 4.13: Temperature vs. Strength Plot of Graphite

CHAPTER 5

CONCLUSION

Graphite shows anomalous Behavior, as its strength increases with increase in temperature as compare to HSLA Steels and Aluminum alloys which are being used in aerospace industry. Due to the anomalous behavior of graphite it can be used for such applications where strength of HSLA Steel and Aluminum Alloys fails. The simple comparison of strength vs. Temperature shows how graphite strength anomalously increase with increase in temperature while steel and aluminum strength decreases. This can clearly see in the plot given below.

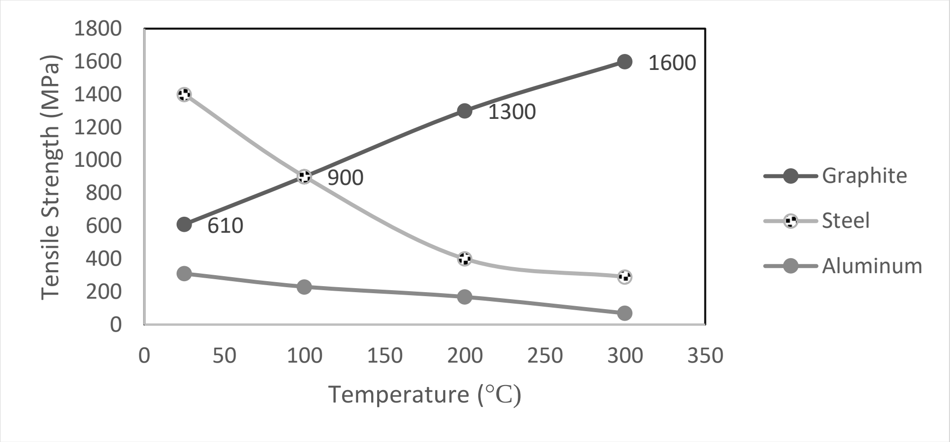


Figure 5.1: Comparison of Strength vs. Temperature of HSLA Steel, Graphite, Aluminum

It is concluded that in severe environmental and oxidative condition (Rocket motor nozzles), graphite/C-C composite is preferable as compare to steel and Aluminum due to its anomalous behavior. Moreover, further enhancements in graphite can be done in order to improve the oxidation resistance of the graphite so that at high temperature oxidation of the graphite can be resisted without affected the strength of the graphite.

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