

Metallurgical Characterization and High-Temperature XRD (HTXRD) Investigation of Fire Resistance Structural Steel

Submitted in partial fulfilment of the requirements for the award of
Bachelor of Engineering in Mechanical Engineering

by

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**DEPARTMENT OF MECHANICAL ENGINEERING
SCHOOL OF MECHANICAL ENGINEERING**

SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**

**Accredited with Grade "A" by NAAC | 12B Status by UGC | Approved by AICTE
JEPPIAAR NAGAR, RAJIV GANDHI SALAI, CHENNAI - 600 119**

April - 2021



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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of SAMUDRALA VENKATA SAI SUHAS (37150165) , PYREDDY VEDA PRANEETHKUMAR REDDY (37150145) who carried out the project entitled “Metallurgical Characterization and High-Temperature XRD (HTXRD) Investigation of Fire Resistance Structural Steel” under my supervision from December 2020 to April 2021.

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We, SAMUDRALA VENKATA SAI SUHAS and PYREDDY VEDA PRANEETHKUMAR REDDY hereby declare that the Project report entitled “Metallurgical Characterization and High-Temperature XRD (HTXRD) Investigation of Fire Resistance Structural Steel” done by me under the guidance of Mr.J.JEYA JEEVAHAN M.E., (PhD.,) is submitted in partial fulfillment of the requirements for the award of Bachelor of Engineering in Mechanical Engineering.

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1.

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ABSTRACT

Fire Resistance Structural Steel is corrosion resistant and highly durable. It is a high-strength low alloy structural steel with high atmospheric corrosion resistance having 0.12% wt. of carbon content. This is used in extreme-pressure applications such as boilers, heat exchangers etcetera. This type of investigation paved the way for the various applications embedded with Fire Resistance Structural Steel which is a better material than the traditional stereotype materials. This was initially developed to challenge the corrosion without any additional painting or layering. The High-Temperature XRD (HTXRD) investigation, XRD (X-Ray diffraction) investigation, Differential thermal analysis (DTA) and, Thermogravimetric analysis (TGA) has given vast detailed information on solid-state, phase transitions, crystallite growth, thermal expansion phase identification, and crystallite size measurement of the of Fire Resistance Structural Steel. The life of this Fire Resistance Structural Steel is more than the usually casted steels. This Fire Resistance Structural Steel has overcome the problem of exhaust manifold failure. The XRD investigation of welded Fire Resistant structural steel is made, and a comparison is drawn among the base metal and, GMAW, GTAW, Laser welded, and AGTAW welded samples. From the investigations, a better weld with transcendent metallurgical properties for Fire Resistant Structural steel is suggested. The results from the investigational study have led to the replacement of tralatitious casted steel with this Fire Resistant Structural steel, which shrinks the frequency of the replenishment of exhaust and chimneys. The D'S pacing [d(ang.)] was almost the same for the sample at all temperatures ranging from 600°C to 1400°C, which was nearly 2.041187 for the highest peak. The vast information from these investigations (graphs and images) were useful in increasing the utilization of this Fire Resistance Structural Steel in chimneys and exhausts in factories and many more high-temperature applications.

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LIST OF SYMBOLS AND ABBEREVIATIONS

AFNOR	Association French Normalization Organization Regulation
AGTAW	Autogenous Tungsten gas arc welding
Al ₂ O ₃	Alumina
Al	Aluminium
ASTM	American Society for Testing and Materials
BCC	Body Centred Cubic
BS	British Standards
C	Carbon
°C	Celsius
CDD	International Centre for Diffraction Data
CECRI	Central Electro Chemical Research Institute
Co	Cobalt
Cr	Chromium
CSIR	Council of Scientific and Industrial Research
CTAC	International Confederation for Thermal Analysis and calorimetry
CT	Computed tomography
Cu	Copper
DIN	Deutsches Institut für Normung (German Institute for Standardisation)
DPS	Dual-phase steel
DSC	Differential scanning calorimetry
DTA	Differential Thermal Analysis
EDS	Energy Dispersive X-ray Spectroscopy
EN	European standards
FCC	Face Centred Cubic
Fe	Iron
FWHM	Full Width Half Maximum

GMAW	Gas Metal Arc Welding
GTAW	Gas Metal arc welding
GTAW	Gas tungsten arc welding
HAZ	Heat affected zone.
HBIS	Hebei Iron and Steel
HCP	Hexagonal Close Packed
HSLA	high strength low alloy
HTXRD	High-Temperature X-Ray Diffraction
IRSM	Indian Railway Service of Mechanical
IS	Indian standard
JCPDS	Joint Committee on Powder Diffraction Standards
JIS	japan Industrial Standard.
K	kelvin
Kv	kilovolt
LCF	Low cycle Fatigue
MgO	Magnesium
MIG	Metal Inert Gas
Min	minute
mm	millimetre
Mn	Manganese
Mo	Molybdenum
MPD	Multipurpose X-Ray Diffraction
Ni	Nickel
nm	nanometre
PARS	Portable Apparatus for Residual Stress
PCM	phase change material
PDF	Powder Diffraction File
PMMA	Polymethyl methacrylate
P	Phosphorus
PSD	Position Sensitive Detector
SDT	Structural Dynamic test

SiC	Carborundum
Si	Silicon
S	Sulphur
TGA	Thermo Gravimetric Analysis
TIG	Tungsten Inert Gas
Ti	Titanium
UNI	Italian National Unification
US	United States
V	Vanadium
WNR	Werkstoffnummer
W	Tungsten
Wt	weight
WZ	Weld zone
XRD	X-Ray Diffraction

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CHAPTER 1

INTRODUCTION

1.1 FIRE RESISTANT STRUCTURAL STEEL

Fire Resistance Structural Steel is corrosion-resistant steel. It was developed by a US company in 1933. It almost took two decades for the material to get popular and reach the public. This is very rarely used around the world and even more rare in India. It is known by various names such as weathering steel, bridge steel, structural steel, and many more. This steel has high durability. It is a high-strength low alloy structural steel and is highly atmospheric corrosion-resistant, having 0.19% wt. of Carbon content and 0.5% wt. of Chromium, which makes the steel resistant against corrosion. Weathering steels are structural low alloying steels with alloying elements such as Ni, Cu, Mo, and Cr making up 3-5% of the overall composition. It has a good anti-corrosion property because it avoids corrosion by creating dense oxide deposits on the surface layers [2]. Fire Resistant Structural Steel's picture is shown in the following **Figure 1.1**.



(Source: <https://www.steelemporium.co.in/corten-steel-pipe-manufacturers-suppliers.html>)

Figure 1.1 Fire Resistant Structural Steel Pipes

This material is very well known for its corrosion resistance properties, it is a more sustainable choice than any other material to overcome the ill effects of corrosion.

This was initially developed to build railroad coal wagons and to challenge the corrosion without any additional painting or layering. This requires a very low maintenance cost. The life of this Fire Resistance Structural Steel is more than the usually casted steels. It develops a rust layer called “PATINA” which shelters the steel from further corrosion.

Inter-critical heat treatment is used to create weathering steels. At an inter critical temperature, tiny groupings of austenite form in the ferrite matrix, that maintain larger compositions of carbon and other alloying elements than the insignificant contents of the steel and thus have a locally upgraded hardenability [11].

These steels will have a corrosion tolerance that is 1–7 times that of high carbon steel coating or galvanizing, which is customarily needed to preserve carbon steel, which can be avoided, resulting in a decrease in self-supporting weight and steel structure maintenance costs. Decades of use and research have proven that these steels have high corrosion resistance in coastal atmospheres (containing Cl^-) as a protective rust coating responsible for the high corrosion resistance has formed after a long time of use, and they have been liberally used in the construction of bridges, transmission towers, homes, guardrails, and lighting poles [25].

1.1.1 Manufacturing Fire Resistant Structural Steel

Smelting (furnace, electric furnace - micro-alloying treatment - Argon-LF refining - low superheat casting - controlled rolling and cooling process route are commonly used for weathering resistant steel. When smelting steel scrap with burden in furnace, according to traditional smelting procedure, after steel tapping added deoxidant and alloy Molten steel by blowing argon and then to cast, after argon is blown, temperature of molten steel by continuous casting machine cast slab the inclusion content of steel purified weathering steel is considerably reduced as a result of the addition of rare earth elements. In the LF refining process, lime and fluorspar slag are used, along with a compound deoxidizer and silicon carbide for diffusion oxygen. The top slag must be white slag before tapping off. Before feeding calcium silicate, soft argon feed processing line after a period of a uniform temperature of molten steel, components, and foster inclusion floating ensure steel quality. Because weathering

steel is made of copper iron, which has a low melting point, the rolling process is a crucial step in the production of weathering steel. To avoid rolling after soaking, we should strictly control the steel temperature burning and furnace atmosphere to avoid unnecessary burning or overheating. To avoid Cu in austenite enriched crystals, billets that produce surface cracks should be scarfed at room temperature, furnace ventilation, the heating temperature control at the lower limit, billets should not be kept at high temperatures within the furnace, and the furnace should be kept in a resurgence or a reducing atmosphere [23].

Rolling links also concentrate on temperature control of key points, cooling speed control, and the ability to control the cooling rate of the rolling process stability [23].

1.1.2 Mechanical properties of Fire Resistant Structural Steel

The typical organisation of weathering steel is pearlite and ferrite; but, after weathering steel hardening, the structure of the steel is composed of ferrite and martensite, with martensite uniformly distributed irregularly like an island on the ferrite grain boundary and the axis of the island parallel to the direction of the ferrite grain boundary. This is due to the fact that during the process of zone heating insulation growth, the original austenite transmutes into martensite under its control. Because diffusion is relatively slow due to the lower heating and keeping temperature in the thermal phase zone, diffusion processes are boundary diffusion-based, thus the higher growth velocity along the austenite grain boundary ferrite, which grew to ferrite low speed, resulting in the formation of irregular, the long axis of the ferrite austenite grain boundaries parallel to the island, thus making the transition from its martensite state also showed this distribution [23].The following **Table 1.1** illustrates the thermal properties of Fire Resistant Structural Steel

Table 1.1 Thermal Properties of Fire Resistant Structural Steel

Thermal Property	Value
Latent Heat of Fusion	260 J/g
Specific Heat Capacity	470 J/kg-K
Thermal Expansion	13 $\mu\text{m/m-K}$

Thermal Property	Value
Thermal Conductivity	44 W/m-K
Melting Completion (Liquidus)	1460 °C
Melting Onset (Solidus)	1410 °C
Maximum Temperature: Mechanical	410 °C

1.1.3 Microstructure of Fire Resistant Structural Steel

Because weathering steel must have adequate strength, ductility, and toughness in addition to excellent corrosion resistance, it must also have the greatest strength and toughness. The yield ratio, strain hardening index n , and plastic strain ratio of weathering resistance steel are all related to formability. Weathering resistance steel with a low yield ratio is required for stamping forming, with the yield ratio of cold rolling steel sheet ideally less than 0.7. To achieve uniform deformation, weathering resistance steel must have a bigger strain hardening exponent n . The two-point technique of determining the tensile curve can be used to determine the value of n . Steel in plastic deformation hardening capacity is represented by the strain hardening exponent, which reflects the uniformity of the deformation process, the forming limit, and whether or not a crack is formed, among other things. When n is enormous, the deformation is uniform. The Plastic Strain Ratio, which is determined through tensile testing, is an important indication of Steel Formability, as it indicates that under the same stress, the vertical deformation between the Thickness and Plate plane directions is distinct [23].

1.1.4 Applications of Fire Resistant Structural Steel

The applications of this steel are not restricted only to rail wagons, but it is also used in the construction of bridges and structures. This is even used in streetlights. In sulfur-rich conditions, these steels are used. These are even used in heavy-duty transport applications. This is utilized in situations that are vulnerable to significant climate change such as train wagons and shipping containers. This is in high demand in the US, Mexico, Canada. As this steel has high durability, high strength, and

minimum maintenance cost involved these are used in the construction of bridges hence the name structural steel. Fire Resistant Structural steel is one such weathering steel. Since the late 1800s, the American Society for Testing and Materials (ASTM) has standardized all grades of steel in the United States [38]. One of the applications is illustrated in the **Figure 1.2**



(Source: <https://www.corten-a-b.com/>)

Figure 1.2 Bridge made of Fire Resistant Structural Steel

This steel in India is graded as IRSM steel which was developed to decrease the rate of corrosion which used to lead to the replacement of railway coaches more frequently. With the development of weathering steel, the velocity of the train has increased, and the corrosion resistance has also increased. Weathering steels are a leading light of high-strength low-alloy steel with microstructures mostly made up of strong martensite particles scattered in a light ferrite matrix [4]. Many pressure vessel makers have introduced Fire Resistance Structural steels for supports and other similar uses. Fire Resistance Structural steel is commonly used in the design of buildings and bridges. To take advantage of its weathering qualities, it is often used unpainted in some applications [26].

Not only in bridges it is also used in artistic sculptures which attract a vast number of people. As this material has many positives and our work may give more information and may lead to an increase in the usage of this material.

1.1.5 Major Manufacturers of The Steel

Some of the leading firms in the global weathering steel industry are companies such as the United States Steel Company (US), Nippon Steel & Sumitomo Metal Corporation (Japan), HBIS Group (China), Tata Steel (India). It is available in two forms namely: painted and unpainted. Fire Resistant Structural Steel Rust stabilization therapies decrease the cost of the life cycle and environmental issues in many applications. TATA Steels is the major manufacturer of this weathering steel in India.

1.1.6 Elemental Composition

The composition of Corten A588 Grade steel is as follows **Table 1.2 Chemical Composition**

Table 1.2 Chemical Composition

Element Symbol	Element Name	% Wt.
S	Sulfur	0.001
V	Vanadium	0.003
Ti	Titanium	0.004
Co	Cobalt	0.006
W	Tungsten	0.01
Al	Aluminum	0.018
Mo	Molybdenum	0.021
P	Phosphorus	0.106
C	Carbon	0.12
Ni	Nickel	0.197
Cu	Copper	0.249
Si	Silicon	0.31
Mn	Manganese	0.36

Element Symbol	Element Name	% Wt.
Cr	Chromium	0.5
Fe	Iron	98.095

1.1.6.1 Effect of Alloying Elements and its melting point

- Iron (Fe) is the element with significant composition in this steel with 98.095%Wt. and having a melting point at 1808.15K this element undergoes a transformation from BCC lattice to FCC lattice [27].
- Carbon (C) is the element with significant composition in this steel with 0.12%Wt. and having a melting point at 3773.15K which is the highest of all other elements present in this steel. The significant carbon properties are, at high temperatures, it binds with oxygen, the property of crystalline structure, and allotropic crystalline forms [27].
- Phosphorus (P) maintains 0.106%Wt. in this steel with a melting point at 317.25K and includes properties such as it does not get dissolved in many liquids, it forms various allotropic forms, and when heated can form phosphides [27].
- Molybdenum(Mo) maintains 0.021%Wt. in this material and holds the melting point at 2890.15K and its properties include these it has the ultimate melting point among all the pure elements, it reacts slowly toward acids with this the strength of steel can be increased at high temperatures, it is one of the very hard transition metals, hardenability, and toughness of quenched steel can be improved with the presence of this element [27].
- Aluminum (Al) maintains only 0.018%Wt. in this steel with the melting point is at 933.12K including properties such as it does not form flakes, Al is soft, lightweight, and is of high strength. It is resistant to oxidation [27].
- Tungsten (W) holds only 0.01%Wt. this steel with a melting point of 3683.15K and has properties such as has high strength, has the highest melting point of all metals. it is resistant to oxidation [27].

- Chromium (Cr) is the other important element in this steel that makes the steel corrosion resistant. has 0.5%Wt., and melting point at 2130.15K. its properties include that it forms a shiny finish, and when this element burns, it forms green chromic oxide [27].
- Manganese (Mn) maintains 0.36% by Wt. in this steel with a melting point of 1518.15K and also includes properties such as it is a hard metal but brittle in nature. It is hard to melt, it reacts with water and is dilute-acid soluble [27].
- Silicon (Si) maintains 0.31%Wt. in this steel and has a melting point at 1683.15K. this element is tetravalent and is very brittle. it has the ability to form hydrides [27].
- Copper (Cu) is another element that holds 0.246% by Wt. and also has a melting point of 1356.15K. It is a ce-centered cubic crystalline structure. This element when exposed to moist air, it forms a greenish film patina [27].
- Cobalt (Co) maintains 0.006%Wt. in this steel with a melting point at 1768.15K. It is a hard ferromagnetic element, brittle in nature. it is stable in air and is unaffected by water.it reacts with dilute acids [27].
- Titanium (Ti) maintains 0.004%Wt. in this steel with a melting point at 1933.15K. It is resistant to corrosion and is a hard and robust element with low density and it can withstand extreme temperatures [27].
- Vanadium (V) maintains 0.003%Wt. in this steel with a melting point at 2163.15K. it is corrosive resistant and even shock and vibration resistant [27].
- Sulfur (S) maintains 0.001%Wt. in this steel with a melting point at 385.95K. it can form many allotropes. it improves machinability and lowers ductility [27].
- Nickel (Ni) maintains 0.197%Wt. in this steel with a melting point at 1726.15K. It is hard and bivalent in nature. it is resistant to corrosion and heat. it is malleable and ductile in nature [27].

1.1.7 Grades Classification of Weathering Steels

This fire-resistant structural steel is being known with different names across different nations, few are as in the following **Figure 1.3**[51]:

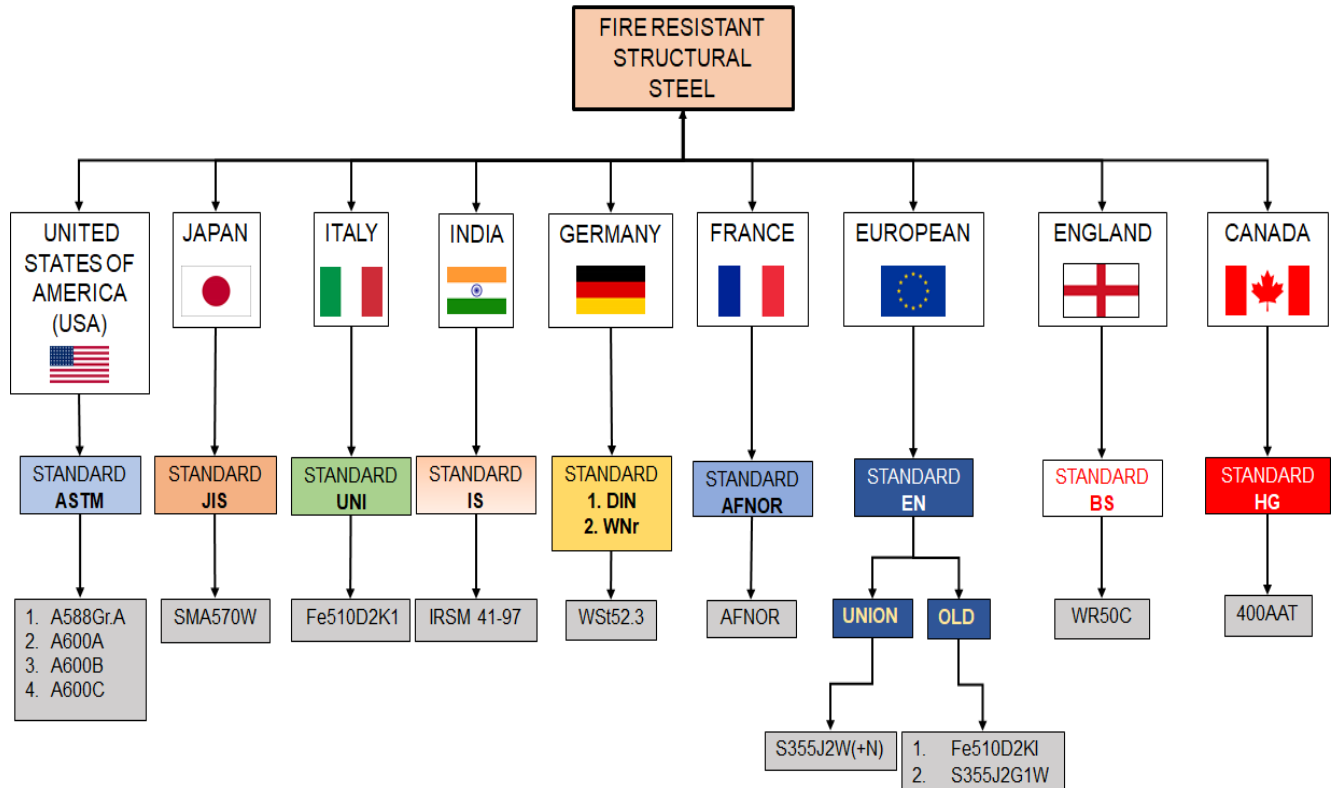


Figure 1.3 Similar grades to Corten A588

The following **Table 1.3** illustrates the different grades of Corten A588 Grade Weathering steel and its elemental composition by weight [34] :

Fire Resistant Structural Steel belongs to Grade A which shows most desirable mechanical and metallurgical properties when compared to that of other Grades.

Table 1.3 Different Grades of Corten Steel

Element	Grade A	Grade B	Grade C	Grade D	Grade E	Grade F	Grade H	Grade J	Grade K
C	0.19 max	0.20max	0.15max	0.10-0.20	0.15max	0.10-0.20	0.20max	0.20max	0.17max
P	0.04	0.04	0.04	0.04	0.04	0.04	0.035	0.04	0.04
Mn	0.80-1.25	0.75-1.35	0.80-1.35	0.75-1.25	1.20max	0.05-1.00	0.035	0.60-1.00	0.50-1.20

Element	Grade A	Grade B	Grade C	Grade D	Grade E	Grade F	Grade H	Grade J	Grade K
S	0.05	0.05	0.05	0.05	0.05	0.05	0.04	0.05	0.05
Si	0.30-0.65	0.15-0.50	0.15-0.40	0.50-0.90	0.30max	0.30max	0.25-0.75	0.50-0.70	0.25-0.50
Ni	0.40max	0.50max	0.35-0.50	----	0.75-1.25	0.40-1.10	0.30-0.60	0.50-0.70	0.40max
Cr	0.40-0.65	0.40-0.70	0.30-0.50	0.50-0.90	----	0.30max	0.10-0.25	----	0.40-0.70
Mo	0.021	----	----	----	0.8-0.25	0.10-0.20	0.15max	----	0.10max
Cu	0.25-0.40	0.20-0.40	0.20-0.50	0.30max	0.50-0.80	0.30-1.00	0.20--0.35	0.30min	0.30-0.50
V	0.20-0.10	0.01-0.10	0.01-0.10	----	0.05max	0.01-0.10	0.02-0.10	----	----
Zr	----	----	----	0.05-0.15	----	----	----	----	----
Co	0.006	----	----	0.04max	----	----	----	----	0.005-0.05
Ti	0.004	----	----	----	----	----	0.005-0.030	0.30-0.050	----

1.2 HTXRD (HIGH-TEMPERATURE X-RAY DIFFRACTION)

High-Temperature XRD (HTXRD) investigation is an investigation more superior, advanced, and even more accurate in giving the information of solid-state, phase transitions, crystallite growth, thermal expansion phase identification, texture analysis, thermal phase transition, lattice dynamics, thermal defect, and crystallite size measurement of material [44]. It is not possible to study and obtain the accurate values of few materials with the traditional way of testing the materials XRD at high temperatures. Traditionally, the material is first heated and then XRD is done this might give values but mostly not appropriate [47].

For any material, the high-temperature phase measurement is either done at room temperature or by quenching the material from high temperature to room temperature[58], but while we cool down the specimen from high temperature to a lower one (room temperature) phase transition might happen and this may disturb our desired results [39]. Such a disturbance can be avoided using this HTXRD (high-temperature XRD) test.

HTXRD (high-temperature XRD) is such a process where the XRD is simultaneously done with the gradual increase in the temperature and the values that occurred are more precise and accurate.

All the phase transitions, crystalline growth, and many more properties are being known at the desired temperatures with more accuracy. Using HTXRD (high-temperature XRD) we can also unveil intermediate phases in a reaction, reaction kinetics, and high-temperature phases that decompose at lower temperatures, these can only be acquired only through HTXRD (high-temperature XRD) while common room temperature scans or tests cannot regulate these results [18]. This test is usually conducted on ceramic materials, pyroelectric materials, ferroelectric materials, magnetic materials, function materials, and superconducting materials [9]. The phase transitions and transmutation in crystallite size with calcination temperatures have been studied in situ using HTXRD [33]. The temperature and time dependences of the phase structure, as well as changes in the lattice parameters, can be determined using in situ high-temperature X-ray diffraction (HTXRD) [58]. The HTXRD graphs are taken and to analyze the phases they are compared with the JCPDS (Joint Committee on Powder Diffraction Standards) of the (ICDD) International Centre for Diffraction Data and remarkable observations are made.

1.3 XRD (X-RAY DIFFRACTION)

Studying the XRD patterns of welded Fire Resistant Structural steel is very advantageous in several applications. X-Ray Diffraction, or XRD for short, is a non-destructive research tool for studying the structure of crystalline materials. XRD analysis is used to classify the crystalline phases present in a substance and thereby reveal chemical composition details by observing the crystal structure [64].

Application and Advantages of XRD include the following as recognizing the various crystalline phases and their orientation in the material, dimensions of the lattice, impact, and scale of grain [1]. XRD patterns of Fire Resistant Structural steels dealt with the exploration of various additional applications. XRD is a fast analytical technique that can provide information on unit cell measurements and is mainly used

for phase identification of crystalline materials. The studied material is thinly ground, homogenized, and the aggregate content is calculated on an average basis [63]. The XRD graphs are taken and to analyze the phases they are compared with the JCPDS (Joint Committee on Powder Diffraction Standards) of the (ICDD) International Centre for Diffraction Data and remarkable observations are made.

In 1912, Max von Laue discovered that crystalline substances function as 3d diffraction gratings for X-ray wavelengths that are close to plane spacing in a crystal lattice. The method of XRD is also widely used to research crystal structures and atomic spacing [29]. Constructive interference of monochromatic X-rays and a crystalline sample is the basis of XRD. A cathode-ray tube produces the X-rays, which are then screened to emit monochromatic radiation, collimated to focus the beam, and guided at the sample. When Bragg's Law ($n\lambda = 2d \sin \theta$) is satisfied, positive interference (and a diffracted ray) results from the association of incident rays with the sample. This law connects the wavelength of electromagnetic radiation to the diffraction angle and lattice spacing in a crystalline sample. Following that, the diffracted X-rays are detected, analyzed, and counted. Due to the arbitrary orientation of the powdered specimen, scanning the sample across a spectrum of 2 angles can include all potential lattice diffraction directions. Since each mineral has its own set of d-spacings, converting the diffraction peaks to d-spacings allows for mineral detection. This is generally done by contrasting d-spacings to regular reference patterns.

The diffracted rays are obtained after X-rays are focused on the sample. The angle between the incident and diffracted rays is a critical component of all diffraction. Beyond that, the instrumentation for powder and single-crystal diffraction differs [29]. An X-ray diffractometer's geometry is such that the sample rotates at an angle in the direction of the collimated X-ray beam, while the X-ray detector is placed on an arm and rotates at an angle of 2θ to absorb the diffracted X-rays. A goniometer is an instrument that is used to rotate the sample and maintain the angle. Data is obtained at 2θ angles ranging from 5° to 70° for standard powder shapes, which are pre-set in the X-ray scan [29].

As the detector and sample rotate around their respective axes, the strength of diffracted X-rays is continuously recorded. When the mineral contains lattice planes with d-spacings suitable for diffracting X-rays at the magnitude of θ , an intensity peak occurs. For higher θ values greater separation anticipates. The center of the diffraction peak at 80 percent peak height is usually used to determine the 2λ direction of the diffraction peak.

Max locations at 2θ and X-ray counts (intensity) are usually displayed in the form of a table or an x-y graph (shown above). Intensity (I) is calculated as either peak height intensity (that is, the intensity above the background) or integrated intensity (that is, the area under the peak). The average of the peak intensity to the most intense peak is used to measure relative intensity.

By solving the Bragg equation for the required value of d , the d-spacing of each peak is determined. Following the determination of all d-spacings, automatic search/match routines equate the ds of unknown materials to those of identified materials. Since each mineral has its own set of d-spacings, matching these d-spacings enables the unknown sample to be found. The d-spacings are ordered in order of intensity, starting with its most intense peak. This is a hierarchical process. The Powder Diffraction File (PDF), which contains d-spacings for hundreds or even thousands of inorganic compounds, is available from the International Centre for Diffraction Data.

XRD tests are performed in instruments called X-ray diffractometers. An X-ray tube, a sample holder, and an X-ray detector are the three fundamental components of an X-ray diffractometer.

In a cathode ray tube, X-rays are created by heating a filament to generate electrons, then applying a voltage to accelerate the electrons into a target and bombarding the target material with electrons. Characteristic X-ray spectra are formed when electrons have enough energy to dislodge inner shell electrons of the target material.

These spectra are made up of multiple elements, the most important of which are $K\alpha$ and $K\beta$. $K\alpha$ is made up in part of $K\alpha_1$ and $K\alpha_2$. $K\alpha_1$ has a much shorter wavelength

than $K\alpha_2$ and is half as bright. The wavelengths used are unique to the target material (Cu, Fe, Mo, Cr) [29].

Filtering with foils or crystal monochromators is essential to obtain monochromatic X-rays for diffraction. Since the wavelengths of $K\alpha_1$ and $K\alpha_2$ are sufficiently similar, a weighted average of the two is used. $CuK\alpha$ radiation = $1.5418\text{\AA}$ is the most typical target material for single-crystal diffraction. These X-rays are collimated before being focused on the sample. The amplitude of the reflected X-rays is recorded as the sample and detector rotate.

This X-ray signal is captured and analyzed by a detector, which then transforms the signal to a count rate, which is then transmitted to a system like a printer or a computer display [29].

Fluids, metals, crystals, polymers, catalysts, fibers, pharmaceuticals, thin-film coatings, ceramics, solar cells, and semiconductors are also studied using XRD, a high-tech, non-destructive method. In several fields, such as microelectronics, power generation, aerospace, and many others, the technique has numerous practical applications. The appearance of defects in a crystal, its resistance to stress, texture, size, and degree of crystallinity, and practically every other variable related to the sample's basic structure can all be easily identified using XRD analysis [1].

One of the most critical conditions for XRD study of powder samples is proper sample preparation. This is particularly valid for soils and clays containing finely divided colloids, which are weak X-ray reflectors, as well as other materials like iron oxide coatings and organic materials, which make XRD characterization more difficult [5].

Micro-XRD (X-ray micro-diffraction) is a non-destructive method for primary phase processing of crystal constituents in sample thickness smaller than 1 mm. It is used to supplement optical and electron microscopy with elemental microanalysis, particularly in complex mixtures with phases with similar chemical compositions. Conversely, scaling up XRD to the microscale by using it on very heterogeneous real samples causes variations from the normal protocol [5].

In mineral discovery, XRD is the most effective method. Since its discovery, mineralogists were among the first to establish and promote the modern discipline of X-ray crystallography. As a consequence, the invention of XRD has practically revolutionized the geological sciences to the point that they are almost impossible without it [5].

Without XRD to unambiguously describe individual crystal structures, any geological community currently engaged in mineralogical studies will be missing today. Each mineral type has a distinct crystal structure that produces a distinct XRD pattern, allowing for rapid recognition of minerals in a rock or soil sample. The XRD details will be used to work out what amounts of the various minerals are present [5].

1.4 THERMO GRAVIMETRIC ANALYSIS (TGA)

Thermo gravimetric analysis (TGA) is one of the thermal analytical methods. It is a method of explaining mass changes under a temperature increase regulated by the program [19]. TGA is used to measure the change in mass loss of weight which includes evaporation, decomposition when the product is in a gaseous state, and even oxidation when the metallic sample is gaining weight [54]. We may obtain information on the thermal stability of the material and its proportion of volatile components by controlling the weight shift that occurs when the sample is heated at a constant rate by performing TGA. TGA measures mass changes in a material as a function of temperature (or time) under a controlled atmosphere [56]. The main objective is to measure the thermal stability and composition of a material. TGA is most useful for dehydration, decomposition, desorption, and oxidation processes.

Three (3) types of TGA exist are shown in the **Figure 1.4**as follows [55]:

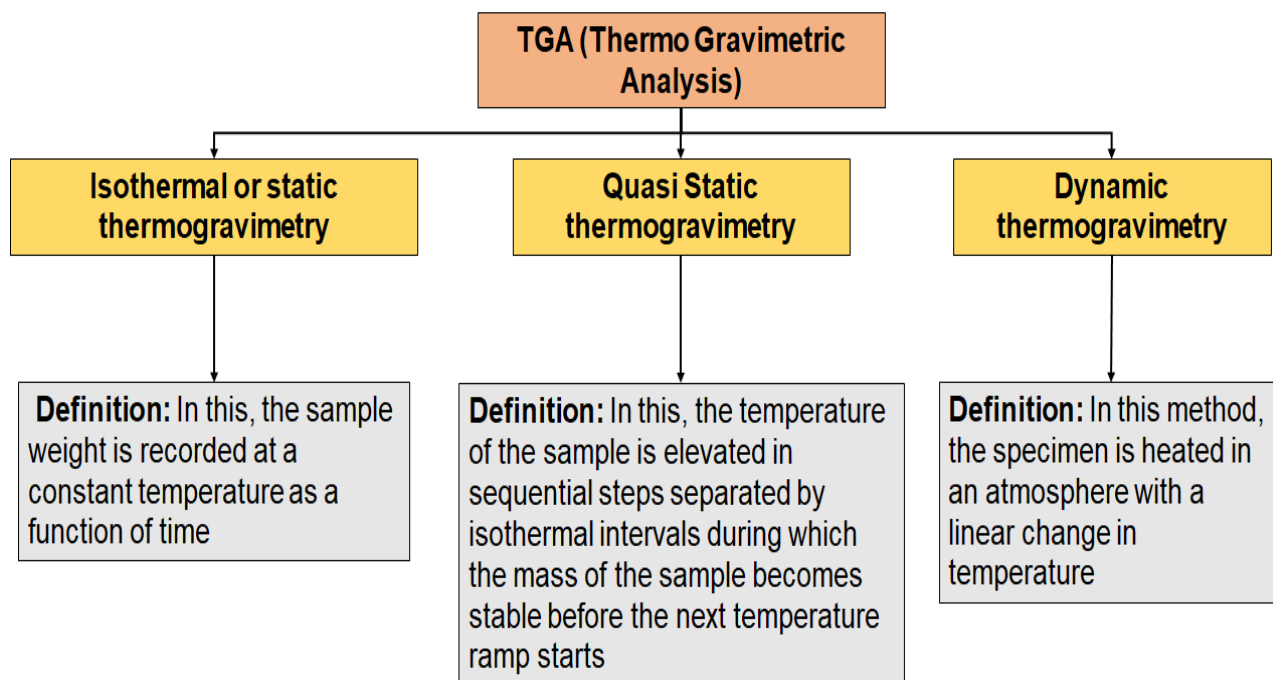


Figure 1.4 Types of TGA

TGA is generally conducted in the thermogravimetric analyzer. A thermogravimetric analyzer measures mass continuously while changing the temperature of the sample over time. The three base measurements in TGA are mass, time, and temperature, any other additional data can be derived from these main base measurements [55]. The thermo - gravimetric data from a thermal reaction is plotted on a graph with the mass or proportion of the original mass on the y axis and temperature or time on the x axis. If the TGA graph heads down we can conclude that there might be a weight loss taken place, whereas if the graph heads upward in the restricted temperature limits, we can conclude that weight gain has taken place. The plot, which is often smoothed, will be the TGA curve. TGA may be used for the study of characteristic decomposition patterns for the classification of materials. Polymeric fabrics such as thermoplastics, thermosets, elastomers, composites, synthetic products, textures, coatings, colors, and fuels are being researched [59].

1.5 Differential Thermal Analysis (DTA)

Differential thermal analysis (DTA) is the most commonly used thermal analytical method. The temperature dependence of the phase transitions is obtained by doing DTA (Differential Thermal Analysis) [3]. DTA gives data on the temperature reactions that happen in the material, whether it is endothermic or exothermic in DTA is described by ICTAC as the technique in which the temperature difference between the sample and the reference material is controlled against time or temperature while the sample temperature is configured in the prescribed atmosphere [37].

Here in DTA the material which is to be tested is compared with reference material, for a particular temperature, the temperature should be the same for both the material until any thermal event occurs, i.e., change in crystal structure or decomposition or melting, etc.[12].

If the temperature in the sample will lag when compared to that of the reference material then the endothermic reaction takes place, if the temperature in the sample exceeds the reference material then the exothermic reaction takes place. The graphs for the endothermic minimum curve and the exothermic maximum curve are observed. This is the information inferred from DTA. A product of the same thermal mass as the sample but with no thermal events across the temperature spectrum of interest is the perfect reference material. In the pharmacy, food industry, cement chemistry, mineralogical research, and environmental sciences, DTA are commonly utilized [19]. For date, bone remains or to research archaeological materials, DTA curves can also be used. Liquid & solidus lines of phase diagrams can be achieved by using DTA [17]. The reference materials for the study of inorganic compounds in DTA are alumina (Al_2O_3), carborundum (SiC), or magnesium oxide (MgO) powder. The enthalpy change is the area below a DTA peak, and it is not affected by the sample's heat capacity [40]

1.6 WELDING

Welding is a fabrication technique used for producing engineering components in various industrial sectors like oil and gas, construction, petrochemical, automobile, food processing, etc. It is the method of connecting metallic components with or without the use of heat, friction, and filler wire. Welding factors include the size and shape of the part to be assembled, the metal framework to be welded, the supply of consumables and equipment, the accuracy required, and the expense of the operation [43]. Welding processes are generally the fusion function by heating the two joined parts together, which may result in material properties being altered or destroyed [50]. The classification of welding can be known in **Figure 1.5**

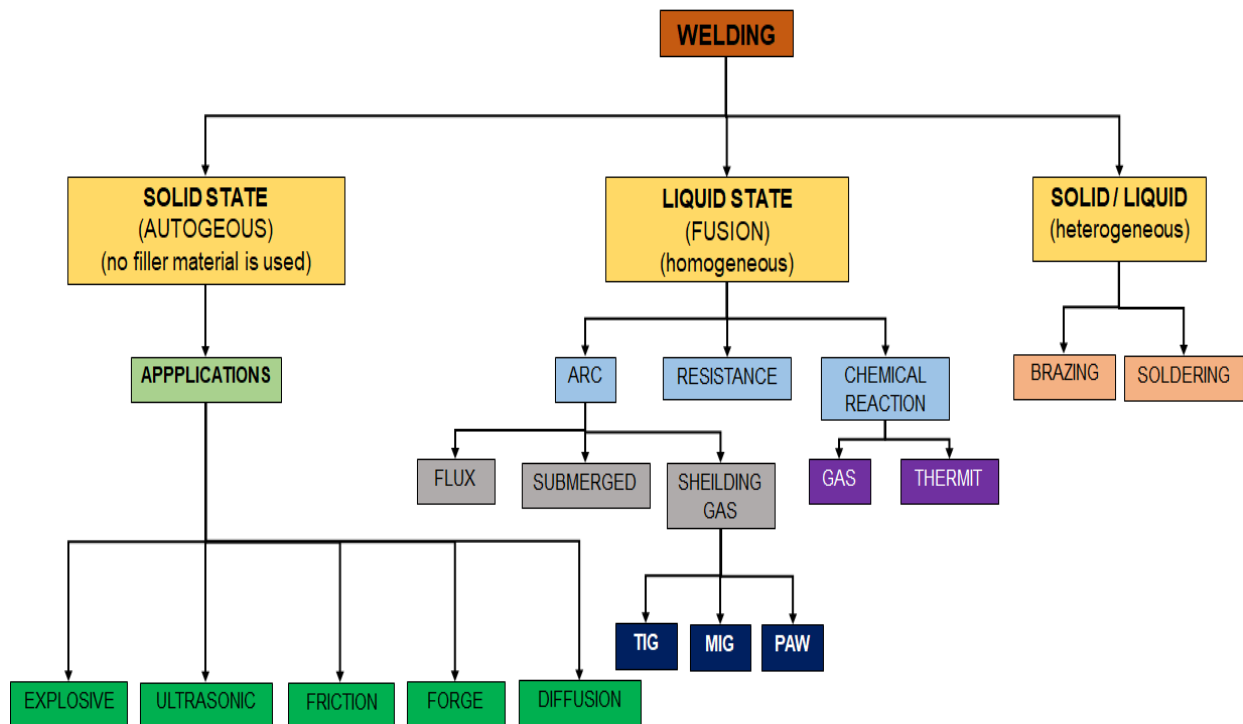


Figure 1.5 Welding Classification

A category of arc welding processes in which a continuous electrode wire is fed into the weld pool by driven feed rolls is known as Gas Metal Arc Welding (GMAW). Between both the wire's tip and the weld pool, an electric arc is created. The wire melts at the same time as it is fed and becomes part of the weld pool. An inert gas

shield prevents both the arc and the weld pool from air exposure and is distributed by a nozzle that is concentric with the welding wire guide tube, this is the basic welding process [43].

The following **Table 1.4** illustrates the welding parameters of four different welds namely GTAW, GMAW, LBW weld and AGTAW Weld.

Table 1.4 Filler material, shielding gas and Heat input during welding.

Type of weld	Filler material	Filler rod dia (mm)	Shielding gas	Heat Input (Q) Joule/mm
GTAW Weld	ER70S-6	1.6	Argon+CO ₂	1338.46
GMAW Weld	ER70S-6	1.6	Argon+CO ₂	1174.34
LBW Weld	No Filler	No Filler	Nitrogen	71.99
AGTAW Weld	No Filler	No Filler	Argon+CO ₂	1332.3

Short-circuiting transfer gas metal arc welding (GMAW), pulsed gas metal arc welding (GMAW-P), gas tungsten arc welding (GTAW), and pulsed GTAW are the powerful welding methods [45]. The most common welding method for Fire Resistant Structural steel is MAG welding. It produces a strong weld with the cheapest welding gas, carbon dioxide. The MAG process originated from the GMAW process, which varies in the usage of shielding gas to protect the weldment from the environment, As compared to welding with stick electrodes, the MAG welding process has a higher output rate. GMAW process gives a high-quality welded joint and also increases production value and product quality [46]. TIG welding is commonly used in several applications because of its accurate material welding, ability to weld a variety of materials, use of less ampere as compared to other welding processes, and clean welding with high control. TIG welding is a little slower than most welding processes [48]. In contrast to MIG and TIG welds, research has found that friction stir (FS) welds are normally better than fusion welds [28]. Avoiding preheating, allowing for cooling to

below 150°C between weld passes, and heat treatment with a minimum temperature rise of 30°C to 50°C to ensure complete dissolution of the intermetallic process in the weldment are the steps taken to obtain high-quality welds [14]. The final product's weld distortions, joint performance, and mechanical properties are influenced by the optimization of TIG welding process parameters. Since the welding process requires a non-uniform heating and cooling process, distortions are inevitable [31]. Welding distortion often creates issues such as spatial inaccuracies during installation and thus component cost rises [13].

Low carbon steels are soft and ductile, and it can be rolled, pounded, sheared, and worked under both hot and cold conditions. When welding low carbon steel, no complications occur. Low carbon steel welds that are perfectly made can be as solid as or stronger than the base metal. They are conveniently machinable and weldable [61]. At room temperature atmosphere, low carbon steel is generally the simplest to weld [42]. Since high carbon steel needs more preheating and cooling after welding than low carbon steel, it is more difficult to weld them. If the carbon counterpart of steel grows, it becomes less weldable [42]. Weathering steels at very high temperatures show a drastic decrease in the ultimate stress, and when cooled down its ultimate stress increases but becomes brittle. As temperatures rose, the decline in σ_u in A588 weathering steel specimens was greater [21].

CHAPTER 2

LITERATURE SURVEY

2.1 HTXRD AND XRD:

(Equihua F et al.,2010) carried out the in situ HTXRD tests on low carbon steel of 0.3 mm thickness. In an Antón Paar high-temperature chamber model HTTK adapted to a Philips X-ray diffractometer model X'PRTMPD with a 40 kV and 55 mA Cu-target X-ray generator, tests were performed.

At 1000 °C Austenite is the only equilibrium phase and the appearance of the ferrite phase is first at 950 °C at 1800 s. The transformation of austenite-ferrite ends at 720 °C 8700 s. $A_{e1}=719$ °C and $A_{e3}=944$ °C are the results from the Calculation of the austenite-ferrite equilibrium temperatures using Thermo-Calc Software AB, 2006 and the complete elemental composition of the experimental steel. 950°C and 720°C are the determined equilibrium temperatures of the A_{e3} and A_{e1} , respectively. The transformation temperatures calculated using Thermo-Calc and estimated using continuous cooling dilatometry show an error of 3% to 5% by the current HTXRD method. At about 44.6° and 43.4°, The Cu K1 peaks of 110 and 111 appear respectively [16].

(Bolt, P. Henk, et al.,2004) carried out HTXRD Experiment on a cold-rolled Ti, Nb-IF steel strip having the following dimensions :(100mmx10mmx1 mm), was placed in Philips X'pert MPD diffractometer, which was equipped with a filtered Cu-K α , radiation source, a Raytech 12° position sensitive detector (PSD), and an Anton Paar HTK2000 heating stage.

In combination with a coil center position, CT of 400-600°C is advantageous for decomposed wustite, which is the desired component for good scale adhesion and cohesion. At first, the only wustite is present, during the start of the tertiary scale which is after secondary descaling. There is a delay for the magnetite to start the development, but once this happens, the fraction of magnetite rises exponentially. If a strip is coiled in the most sensitive temperature area above 400 ° C, no wustite can be

preserved. Large quantities of hematite are produced at high temperatures near the edges of coils coiled ($CT > 500^{\circ}\text{C}$). Very low to very high CTs are harmful to the stability of the scale due to high fractions of retained wustite. decomposition of Wustite into magnetite and ferrite proceeds rather slowly just below 570°C , accelerates with a decrease in temperature, and becomes rapid at around 400°C . when the temperature is reduced further, it slows down but does not stop until the temperature is below 300°C . Wustite diffraction peak splitting and/or shifting during HTXRD experiments demonstrated the occurrence of wustite crystals with varying Fe/O ratios [36].

(Gu, X. Y et al., 2015) Made research on The XRD analysis of MAG welded S355J2W hot rolled steel plate of thickness less than 10mm which resulted in a structure of α -Fe in the joint, the transformation of α (ferrite) $\rightarrow$ γ (austenite) occurs during the third pass welding period where peak temperature is between melting temperature and 1100°C . during the healing process, austenite observed showed no growth when peak temperature ranged from 1100 to A_3 , where A_3 stands for the critical temperature of transformation between ferrite and austenite. At a peak temperature ranging from A_3 to 1100°C The normalized zone obtains. during heating, The growth of austenite ceases [62].

(Hadianfard, M. J et al., 2007) Performed the XRD analysis on a 3mm thick Dual-phase steel plate (DPSs) which is low carbon steel consisting of martensite and ferrite, using a Bruker D8 Advance Diffractometer with 2 ranges 10° to 120° , Interrupted LCF specimens were tested. for measuring peak broadening $\text{Cu K}\alpha$ and (1 0 0) plane was used, XRD measurement results show that XRD peak profiles of (1 0 0) planes for the virgin specimen and LCF specimens tested at a cumulative strain amplitude of ± 0.1 after interruption at 10%, 20%, 50% and 75% of their lifetime. Analyses of XRD peak broadening indicate that FWHM is sensitive to the stage of LCF fatigue life of the materials [29].

(Sulaiman et al.,2018) Has performed The XRD pattern on the sample, observed at room temperature in the range of 2θ between 30° and 70° which matched the ICDD card# 60696 for α -iron. XRD peaks were identified at $2\theta = 44.59^\circ$, and 65° [52].

(Allam Tarek et al.,2015) performed XRD test on the dual-phase weathering steel plate which has a thickness of 3mm. The test leads to the observation that the rust layer formed on the surface of the specimens mainly consisted of Fe_3O_4 and Fe_2O_3 phases. for metallic iron (Fe) a peak in the XRD pattern can be observed. From the XRD patterns, the peak formation is mostly done with the formation of Fe_2O_3 when compared to that of Fe_3O_4 [53].

(Shu Zhen et al.,2014) performed research on the 16 mm thick plate weathering steel of standard S355J2W+N, with hybrid laser-MAG welding and XRD test was done. The weld zone phase composition mainly consisted of α -Fe. It was apparent that the weld zone microstructure consisted largely of columnar ferrite, acicular ferrite, lump ferrite, and a small volume of pearlite. The peak formation with α -Fe is observed at $44.5^\circ, 65^\circ, 83^\circ, 99^\circ$, and 116° with intensities as 1500,200,300,100,120 respectively. Out of which the highest peak is noted at 44.5° at an intensity of 1500 [50].

(P. Dhaiveegan et al.,2016) Performed XRD test on weathering steel plate of 3mm thickness on its corroded layers was obtained using a PANalyticalX'Pert PRO diffractometer in the range of 10° - 70° at a step size of $5^\circ/\text{min}$ using X-ray Source as $\text{CuK}\alpha 1$ and the following observations took place:

The XRD graphs conclude that the presence of iron oxides such as maghemite (γ - Fe_2O_3), magnetite (Fe_3O_4) and hematite (α - Fe_2O_3) and oxyhydroxides such as lepidocrocite (γ - FeOOH), goethite (α - FeOOH), akageneite (β - FeOOH) and ferrihydrite (β - FeOOH) was formed with varying intensities on the rust layer. At 21° , the presence of high amounts of sulfur in the earthward rust layer makes the skyward layer's relative peak intensity stronger than goethite. At 14° the skyward surface is much weaker than the EOL relative peak intensity of lepidocrocite [35].

(Jun Yan et al.,2010) Performed XRD test on TIG, laser, and TIG-laser welded 304-grade stainless steel plate of 3mm thickness in Philips X-ray diffractometer and following observations were made:

All three welded regions had a d-Fe phase and g-Fe phase. The diffraction peak of d (110) has the highest intensity in the JL joint, whereas the diffraction peak of d(110) has the lowest intensity in the JT joint. The d-Fe content is higher in JL and JH joints when compared to that of the JT joint [24].

(Lee. W.B et al.,2009) Carried out an XRD test on GMA welded Dual-phase steel plate of 2 mm thickness. The samples were exposed to the atmosphere and corroded layers were taken under consideration. The observations made are as follows, the exposure time is directly proportional to the peak broadening, as the exposure time increases the peak broadening increases. Grain refinement and cumulative plastic deformation within the grain are responsible for this peak broadening. This makes the region crystalline [60].

(Cunningham David R., 1994) Carried out XRD research on GMA welded HSLA (high strength low alloy) steel plate and remarkable observations are made. The residual stress data were collected using a TEC model 1610 Portable Apparatus for Residual Stress (PARS) vanadium filtered chromium radiation of wavelength 2.290920 Å diffracted from the (2 1 1) planes of iron at $156^\circ 2\theta$. Although the diffracted intensities associated with high 2θ angles are not as large as those associated with lower 2θ angles, changes in 2θ are simpler to discern at high 2θ angles [15].

(Xinge Zhang et al., 2018) Performed XRD test on the GMA spot welded 2mm thick plate of CR590T/340YDP galvanized dual-phase steel plate using the D/MAX-2500PC, Rigaku X-ray diffractometer (XRD) machine with the following parameters 50kV voltage, 300 mA current, Cu K α radiation, and 4°/min scanning. With reference to the XRD pattern, the base metal was majorly composed of ferrite with 86.2 % and 13.8 % of martensite [63].

(Shipin Wu et al., 2019) Carried out XRD test on the MIG welded martensite-austenite dual-phase steel plate of thickness 0.5 mm in D8 Advanced with the voltage 40 kV, current 40 mA, and Cu-K α radiation with a phase period of 0.02° from 30 to 120 degrees. Prior to the XRD test, the specimens were electro-polished with a solution of 95% alcohol and 5% perchloric acid. The volume fraction of austenite for the AW, S-ART, and C-ART deposited metals is 13 percent 22 percent, and 17 percent, respectively, according to the XRD trends of all deposited metals. The dislocation density of martensite in the deposited met was determined using the MWH process. S-ART and C-ART deposited metals have dislocation densities of 0.251015 m⁻² and 0.791015 m⁻², respectively [49].

(Ali Waqas et al., 2019) Performed the XRD test using X'Pert PRO with a copper anode and generator settings with the power supply 40 mA and 40 KY on a GMA welded low carbon steel plate of 5 mm thickness. The existence of a ferritic structure of predominantly pure iron in the central part of the former wall is verified through XRD analysis. The peaks are formed at 45°,65°,82.5° with intensities at 5500,1500,2000 respectively [5].

(Zakaria Boumerzoug et al., 2010) Carried out XRD test on MIG welded low carbon steel plate, Cu-K α radiation was used to evaluate the primary phases of welded steel. XRD was done on the welded HAZ (heat affected zone). Only ferrite phase was observed from the XRD results considering the three peaks which subsequently showed ferrite peaks: bcc (110), bcc (200), and bcc (111) [65].

(Lima. C.R.C et al., 2015) performed X-ray diffraction on GMA welded Low carbon steel of 0.8 mm thickness plate using Shimadzu equipment (Cu, where the wavelength is (λ) = 1.54 nm). The welded specimen has a low proportion of austenitic process, according to the X-ray diffraction pattern [10].

(Ravindra Kumar et al., 2016) carried out XRD analysis directly on the PC-GTA-welded substrate surface having 2mm thickness, with Cu-K α radiation at 2/min scan rate within the scan angle between $20^\circ < 2\theta < 110^\circ$ and different phases were analyzed on the low carbon steel the peaks were formed 45° at an intensity 88 with ferrite formation being observed, another peak was formed at 82° at an intensity 37 with the iron carbide formation being observed. The next major peak was observed at 94° at an intensity of 30 and Fe₂C is formed [41].

(Mahmud SarkariKhorrami et al., 2014) Performed X-ray diffraction (XRD) research with the usage of Cu-K α irradiation on GTA welded Low Carbon Steel of 2mm thickness.

The constituent ferrite phase was consistent as observed from XRD results. For the filler-added specimen, the presence of characteristic peaks (indications for (0 0 2) and (3 0 1) planes) indicates martensite formation.; For AGTAW weld wire, however, only ferrite characteristic peaks occur. Martensite deposition in the weld metal and HAZ of ferritic stainless steels with low and medium chromium is a natural occurrence. Considering the low carbon content of the base and filler metals used in this analysis, the transition directly from liquid to room temperature in weld metal tends to follow: (Liquid) $\rightarrow$ (Liquid + Ferrite) $\rightarrow$ (Ferrite) $\rightarrow$ (Ferrite + Austenite) $\rightarrow$ (Ferrite + Martensite). As compared to the other direction, this one results in a lower volume fraction of martensite inside the fusion zone (i.e., austenite cannot occur in both liquid and ferrite during solidification). (Liquid) $\rightarrow$ (Liquid + Ferrite) $\rightarrow$ (Liquid + Ferrite + Austenite) $\rightarrow$ (Ferrite + Austenite) $\rightarrow$ (Ferrite + Martensite) This path leads to a greater abundance of martensite [32].

(B.-S. Huang et al., 2016) Performed the X-ray diffraction (XRD) tests on GTA welded Low Carbon Steel plate of 3mm thickness.

Samples were sliced orthogonal to the weld seam, subsequently, metallographic processes were used, culminating in 5000Cw waterproof abrasive paper milling and 12000Cw polishing. The WM was subjected to XRD examination. The pattern shows that phase, α phase, Cr₂Ni₃, and FeNi₃ are present in the WM, but σ phase and M₂₃C₆

(chromium carbide) are not present, indicating that the nature of the WM is excellent [6].

(M. Mosallae pour et al., 2011) Performed the XRD test on GTA welded Low Carbon Steel Plate of thickness 3mm. XRD experiment with Cu- α radiation in 2θ range from 10° to 120° with $0.04^\circ/\text{second}$ angle rate was used to analyze the process of the clad constituent.

The phase composition of the clad layer was analyzed using the XRD pattern of samples, which revealed the formation of approximately 65 percent FeAl phase, about 34 percent Fe_3Al phase, and 3 percent $\text{Fe}(\alpha)$ phase in the structure of the clad layer. The FeAl intermetallic was used as the matrix for the overlay clad. The FeAl_3 components are developed in the clad layer, far from the clad/substrate interface, according to XRD research [30].

(Jie H. Chen et al ., 2009) Performed the X-ray diffraction (XRD) analysis on GTA welded Low Carbon Steel of 4mm thickness and the crystalline structure of the sample is identified, with Cu- α radiation in 2θ ranging from 20° to 100° .

According to measurements based on Bragg's theorem, the lattice constant of the BCC phase is 0.286 nm, and the FeMoSi phase is an HCP structure with $a = 0.765$ nm and $c = 0.477$ nm. However, a BCC phase order peak was discovered at $2\theta = 31$. The FeMoSi process with a multicomponent is contained in the dendrite rich in Mo, Fe, and Si, whereas the BCC phase is found in the interdendrite. Since the interdendrite is rich in Al, and Al is a stronger BCC phase formed in multicomponent alloy systems, it is fair to assume it's made up of a BCC solid solution phase [22].

2.2 TGA AND DTA:

(Sulaiman et al.,2018) carried out TGA on ultra-low carbon steel, using Perkin Elmer TG/DTA unit, where the temperature ranges from 30° to 1200°C with a heating/cooling rate of $10^\circ\text{C}/\text{min}$.

The observation states that there was a minor wt. % loss (~0.73 wt. %) in the 30°-785°C temperature range. which might have occurred due to the removal of other oxides or entrapped surficial water. Later, the sample in the temperature range of 785°-1200 °C, with a gain rate of about 3.2×10^{-3} mg/K has gained an extra weight (1.31 wt. %) which might have formed due to the formation of nitrides as a result of chemical reaction of nitrogen, that's been used as a purging gas. Performed energy-dispersive X-ray spectroscopy to obtain the elemental composition. this resulted in detecting mainly iron as a major component with a small amount of carbon, aluminum, manganese, and oxygen as impurities. From the results, it is stated that iron is 96.63% inWt. % and 89.95% in atomic %. Whereas carbon has 0.76% by weight and 3.26% atomic % [52].

(Cheng Jiaji et al., 2020) Has performed TGA on the phase change material (PCM) microcapsule and from the TGA graphs.

The following observation was made that between 130° and 300 °C, the first part of weight loss occurs because the impurities decompose and volatilize in PMMA. the breakdown of PMMA begins at about 300°C. The TG curves of Ref-PCMs and C-PCMs begin to decrease from about 165°C which indicates that there is a loss of weight and the curves tend to be stable at 250°C and 270°C, this indicates that there is no major loss in weight of the component. This is because the n-octadecane present in the microencapsulation gasifies and destroys the shell. At the end of the test, the PMMA will continue to decompose. The C-PCMs' residual mass (4.5%) is larger than that of Ref. PCMs (1 %), as CNTs do not decompose below 600 C in the C-PCMs [20].

(P. Dhaiveegan et al., 2016) Carried out TGA test on the weathering steel plate of 3mm thickness on its corroded layer in TA SDT Q600 thermal analyzer at a heating rate of 5°C/min from 30°C to 1000°C in an air atmosphere and the following observations took place:

The observation revealed that the weight loss of iron oxyhydroxides and oxides for skyward (layer exposed to the air) and earthward (layer exposed to the ground) rust layers are distinct at different temperatures .in the temperature range of 20° to 220° C rapid weight loss occurred this is due to the desorption of physiosorbed water molecules. the next weight loss occurred at the temperature 370° C this was resulted because of the accelerated transformation of oxyhydroxides to iron oxides .Performed energy-dispersive X-ray spectroscopy to obtain the elemental composition using Quanta 200 FEG and the major observation made was that there were chloride, sulfur, and exogenous silica with alloying elements in the rust layers the layer exposed to the sky has Cr, Mn, and Ni more in content, whereas the surface exposed to earth has enriched only with Ni , the earthward surface has more chloride content when compared to that of the skyward surface, But is completely opposite when it is about the sulfur content [35].

(Sulaiman et al., 2018) has carried out DTA on ultra-low carbon steel, using Perkin Elmer TG/DTA unit, in the temperature range of 30-1200 °C with a heating/cooling rate of 10 °C/min.

While the temperature increased up to 500°C the DTA curve showed a downward slope.the sample in which the FeC₃ cementite present gets dissolved in ferrite iron which is a BCC crystal having magnetic properties up to ~770 °C. The material shows an exothermic behavior at a temperature above 500°C and a small peak was observed at 727°C stating that above this temperature the ferrite iron converts into two-phase ($\alpha + \gamma$) iron but still maintains its BCC crystal structure.A rapid rise at 910 °C was also observed, revealing that the ($\alpha + \gamma$) iron undergoes a phase transition from two-phase (BCC) structural iron to single-phase (FCC) γ -iron, called austenite, beyond this temperature [52].

(Smetana. B et al., 2012) Performed DTA on micro-alloyed steels DTA curves are obtained for the analyzed alloys at the heating rate of 7 °C·min⁻¹.

The liquidus line of the Fe-C diagram is higher than the liquidus temperatures. liquidus and solidus lines of the Fe-Mn system are higher than the experimental

temperatures of solidus and liquidus. In some instances, substantial variations in the small temperature interval for the γ - δ transition were found in the experimental results and they are compared with theoretical measurements. The width of this interval is mainly influenced by Carbon. 48 °C and 41 °C are the temperature interval of the samples [8].

CHAPTER 3

AIM AND SCOPE

3.1 AIM

- To investigate up to what temperature the Fire Resistance Structural Steel can withstand.
- To know how Fire Resistance Structural Steel is used at very high temperatures.
- To understand whether using this material in chimneys functioning at very high temperatures is feasible.
- To obtain the characteristic data of Fire Resistance Structural Steel at very high temperatures and its respective applications.
- To perform High Temperature XRD (HTXRD) investigation so that information of solid state, phase transitions, crystallite growth, thermal expansion phase identification, texture analysis, and crystallite size measurement of Fire Resistance Structural Steel can be obtained.
- To perform XRD (X-Ray diffraction) analysis so that information of the crystalline structure of Fire Resistance Structural Steel after heating can be obtained.
- To perform Differential thermal analysis (DTA) which leads in obtaining the information of the temperature dependence of the phase transitions of Fire Resistance Structural Steel.
- To perform Thermogravimetric analysis (TGA) which leads in obtaining the information of Fire Resistance Structural Steel's thermal stability and its fraction of volatile components by monitoring the weight change that occurs as the sample is heated at a constant rate.
- To perform XRD investigation on GTAW, GMAW, LBW welded and AGTAW welded samples and a better weld for Fire Resistant Structural steel is suggested.

3.2 SCOPE

- The outcome will be a detailed report of all the characteristic properties of Fire Resistance Structural Steel with values from High Temperature XRD (HTXRD) investigation, XRD (X-Ray diffraction) investigation, Differential thermal analysis (DTA) , and Thermogravimetric analysis (TGA). The data obtained through above analyses will be useful to all the factories and industries which deal with low carbon steel.
- The scope is to replace traditional steel chimneys which do not last for a long time by this Fire Resistant Structural steel, which has high durability, and can withstand high temperatures.
- The outcome will be the advantages of Fire Resistance Structural Steel in the field of high temperature applications.
- The above given analyses will fetch a huge amount of information in knowing the Fire Resistance Structural Steel applications better.
- A comparison between XRD graphs of different welded Fire Resistant Structural steel is done and a better weld is recommended.

CHAPTER 4

METHODOLOGY AND EXPERIMENTAL SETUP

4.1 METHODOLOGY

The methodology of the tests has been displayed in the following **Figure 4.1**

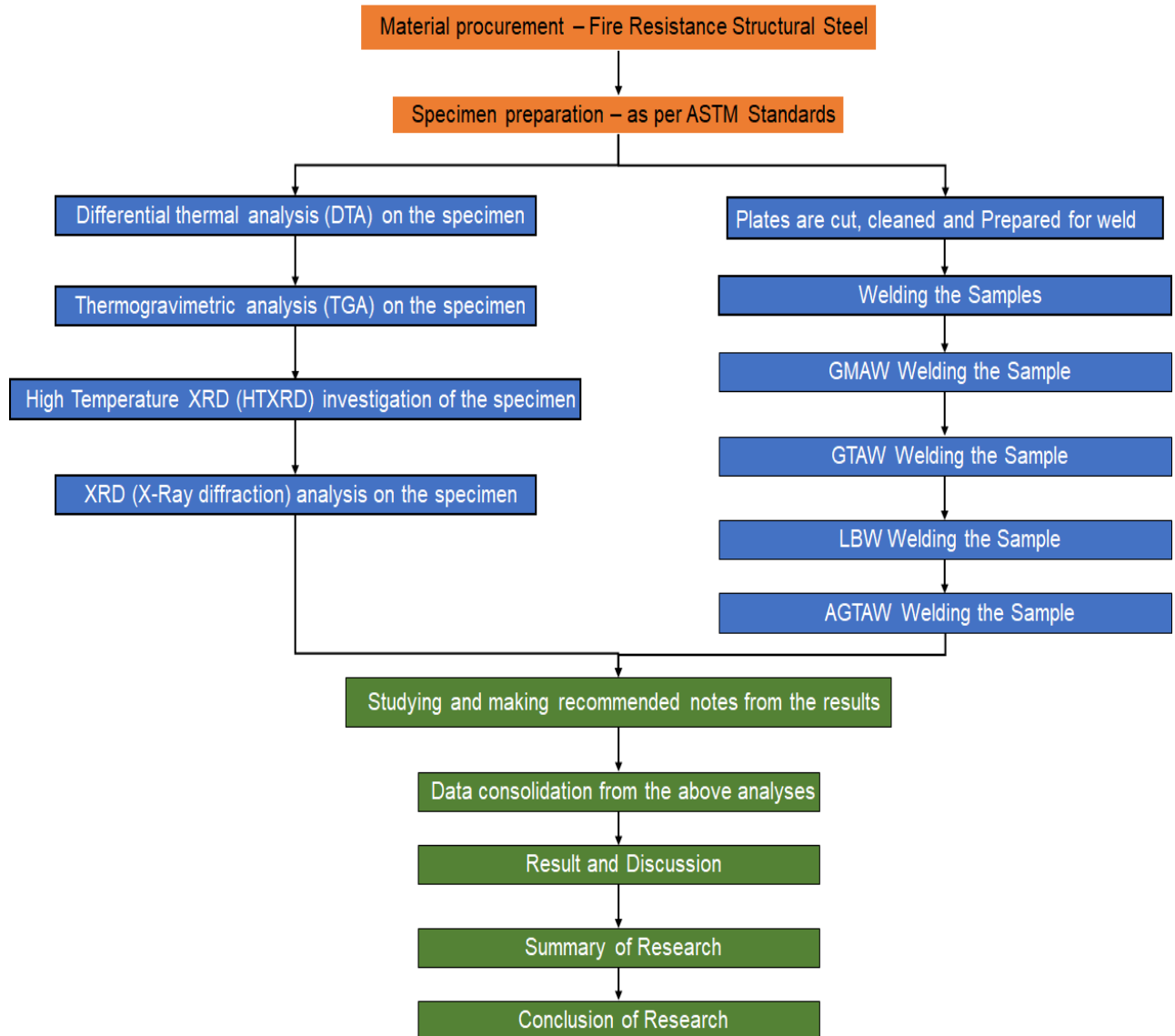


Figure 4.1 Stages in research

Firstly, Corten A588 Grade Steel sheet was purchased from Sharoff Forgings Private Limited. And then it is cut down into respective sizes to undergo the following tests

HTXRD (High Temperature X-Ray Diffraction), XRD (X-Ray Diffraction), DTA (Differential Thermal Analysis), and TGA (Thermo Gravimetric Analysis) and respective results were noted down. later the other samples were welded, by four different welding techniques namely, GMAW(Gas Metal Arc Welding), GTAW (Gas Tungsten Arc Welding), LBW (Laser Beam Welding) and AGTAW (Autogenous Gas Tungsten Arc Welding) and XRD (X-Ray Diffraction) analysis were performed. from all these experimental data, data consolidation was done, and a report was prepared.

For the experimental proceeding of HTXRD test, the samples were sent to Central Electro Chemical Research Institute (CECRI) which is a chain of CSIR (Council of Scientific & Industrial Research). And the HTXRD test was performed there.

4.2 SPECIMEN PREPARATION

Prior to the tests the samples of Fire Resistant Structural Steel were cut into dimensions of (10 * 10 * 0.8 mm). The thickness as mentioned is less than 1 mm. The rust layer, (known as patina) is removed using a 140-grade emery paper as shown in **Figure 4.4** and the AutoCAD diagram is shown in **Figure 4.3** is polished till it has obtained a shiny finish with a soft wheel. It is then washed with acetone and made to dry in the air, the specimens for inspection were prepared according to ASTM specifications. After going through the above-mentioned process, the samples are further processed to their testing stage. These samples as shown in **Figure 4.2** were later used for HTXRD (High Temperature X-Ray Diffraction), XRD (X-Ray Diffraction), DTA (Differential Thermal Analysis), TGA (Thermo Gravimetric Analysis), and remarkable results and conclusions were noted down.



Figure 4.2 XRD sample

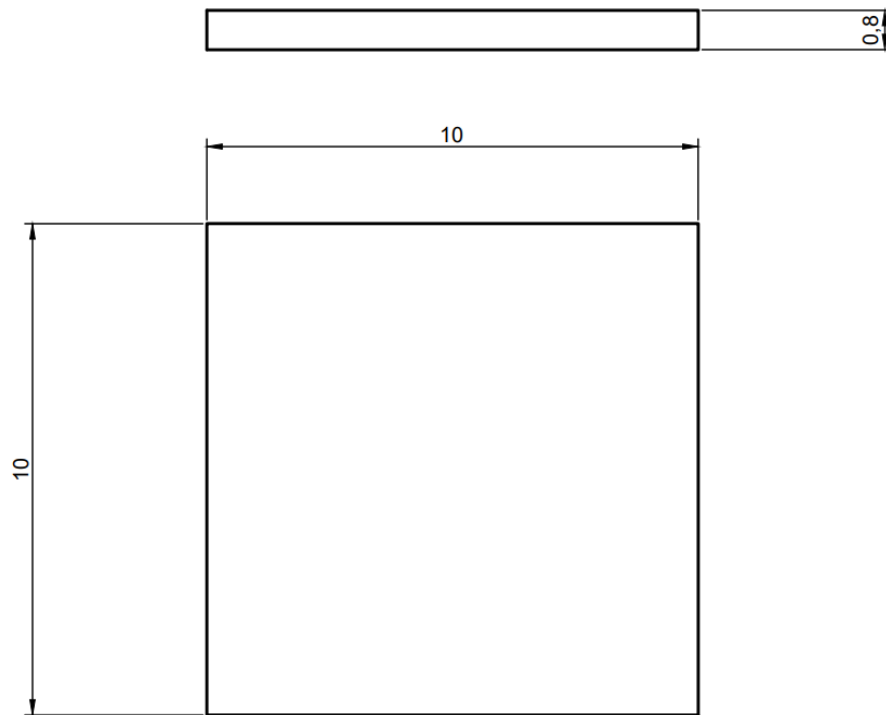


Figure 4.3 AutoCAD diagram of the Specimen

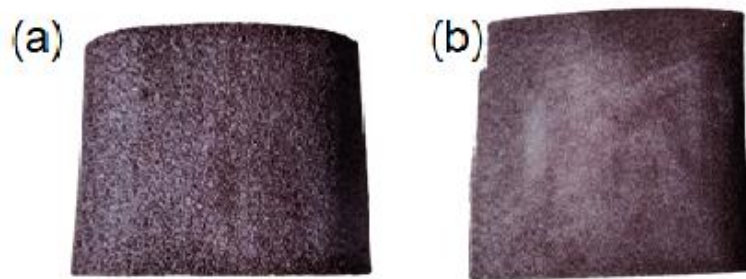


Figure 4.4 Emery Paper (a) and Emery Paper (b)

4.2.1 HTXRD (High Temperature X-Ray Diffraction)

The High temperature XRD test was carried out from the temperature ranging from 600°C to 1400°C in the Rigaku Ultima IV machine as shown in the **Figure 4.5** below. For this test the sample was made to come a thickness less than 1mm.



Figure 4.5 HTXRD (Rigaku Ultima IV) Machine

4.2.2 TGA (Thermo Gravimetric Analysis)

TGA analysis was made on the sample at two different temperature ranges in the TG-DSC machine as shown in the **Figure 4.6**

- Room temperature to 1000° C
- Room temperature to 1400° C

4.2.3 DTA (Differential Thermal Analysis)

DTA analysis was made on the sample at two different temperature ranges.

- Room temperature to 1000° C
- Room temperature to 1400° C



Figure 4.6 TG-DSC Machine

4.3 SPECIMEN PREPARATION FOR WELDED

Preceding to welding of the Fire Resistant Structural steel of thickness of 2mm, the rust layer, (known as patina) is removed using a 120-grade emery wheel and is polished till it has obtained a shiny finish with a soft wheel. It is then washed with acetone and made to dry in the air, the specimens for inspection were prepared according to ASTM specifications after the plates were welded. XRD (X-Ray Diffraction) test was carried out on the welded Fire Resistant Structural Steel. Four samples were taken, and each was welded with four different welds such as GTAW, GMAW, LBW weld, and AGTAW weld. These four samples were compared with the base metal and conclusions are noted down.

4.3.1 Welding

Fire Resistant Structural Steel is taken and is cut into the dimensions of (20*20*2mm). This way five samples were made. The first sample was made to undergo GMAW where the filler rod is ER70S-6, and the shielding gas used was 80%CO₂ and 20%Ar. The next sample was made to undergo GTAW, here the filler rod is ER70S-6, and the shielding gas used is Argon or Helium. The shielding gases and filler rod are only used in GTAW and GMAW, these give additional protection to the weld. The third sample was made to undergo Laser welding, where the laser used was CO₂, here neither the filler rod nor the shielding gas is used. The next sample of the Fire

Resistant Structural steel was made to undergo Autogenous gas tungsten arc welding (AGTAW), where there was no usage of filler material, in this type of weld the base material is heated and melted, and then the weld was formed. with the four welded samples and one non welded sample of Fire Resistant Structural steel grade is considered for the XRD (X-Ray Diffraction) analysis. The XRD analysis of Fire Resistant Structural steel base material and all-welded plates was performed to classify the different phases developed in the weld zone (WZ).

CHAPTER 5

RESULT AND DISCUSSION

5.1 HTXRD (HIGH TEMPERATURE X-RAY DIFFRACTION)

When the test was made ranging from 600°C to 1400°C, remarkable changes can be noticed at room temperature (that is 25°C), 600°C, and 1300° C and all the graphs and peak formations are compared.

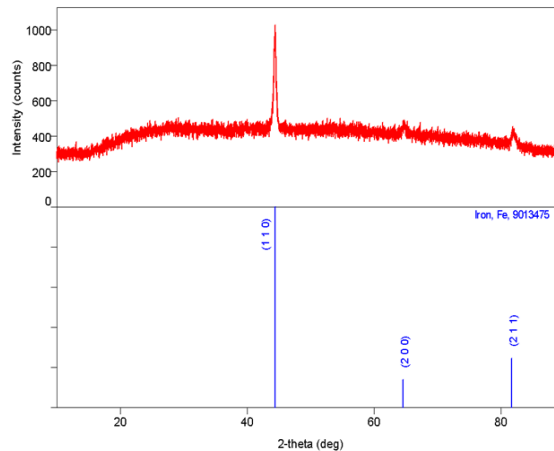


Figure 5.1 HTXRD graph at Room Temperature

The above pattern in the **Figure 5.1** is the XRD pattern of Fire resistant Structural Steel at room temperature i.e. 25°C and the following inference can be made: The remarkable peaks are formed at 44.397358°, 64.652341°, 81.999375°, from the data of JCPDS (Joint Committee on Powder Diffraction Standards) of The (ICDD) International Centre for Diffraction Data with respective intensities at 100, 20 and 30, α -Ferrite formation can be noticed. The phase names are Iron (1,1,0), Iron (2,0,0), Iron (2,1,1) respectively. the crystalline size at 44.397358° is 4.996925, while the crystalline size at 64.652341° is 13.228839.

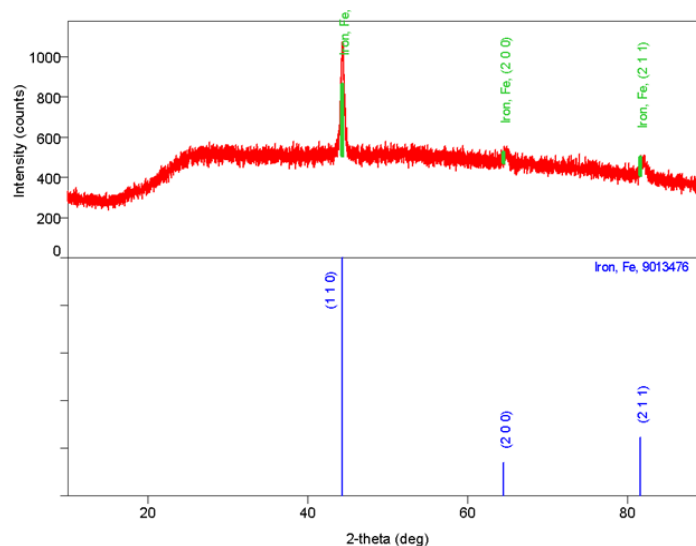


Figure 5.2 HTXRD graph at 600°C

The above pattern in the **Figure 5.2** is the HTXRD pattern of Fire Resistant Structural Steel at 600°C and the following inference can be made: The remarkable peaks are formed at 44.34257°, 64.661665°, 82.085°, from the data of JCPDS (Joint Committee on Powder Diffraction Standards) of The (ICDD) International Centre for Diffraction Data with respective intensities at 100, 20 and 30, α -Ferrite formation can be noticed. The phase names are Iron (1,1,0), Iron (2,0,0), Iron (2,1,1) respectively. The crystalline size at 44.34257° is 3.87363 and the crystalline size at 64.661665° is 21.163232, while the crystalline size at 82.085° is 0.

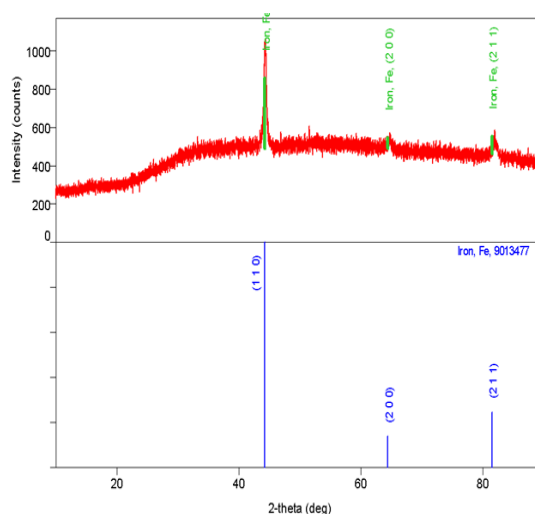


Figure 5.3 HTXRD graph at 1300°C

The above pattern in the **Figure 5.3** is the HTXRD pattern of Fire Resistant Structural Steel at 1300°C and the following inference can be made: The remarkable peaks are formed at 44.307633°, 64.832375°, 81.99775°, from the data of JCPDS (Joint Committee on Powder Diffraction Standards) of The (ICDD) International Centre for Diffraction Data with respective intensities at 100,20 and 30, α -Ferrite formation can be noticed. The phase names are Iron(1,1,0), Iron(2,0,0), Iron(2,1,1) respectively. The crystalline size at 44.307633° is 4.810318, whereas for the other two peaks the crystalline size of the sample is 0.

Table 5.1 HTXRD data

Sample Before Heating							
2-theta (deg)	Esd (2-θ)	D (ang.)	FWHM (deg)	Esd (FWHM)	Crystallite size(ang.)	Esd (Crystallite size)	Phase name
44.397358	0.010837	2.038795	0.399471	0.008898	224.335816	4.996925	Iron(1,1,0)
64.652341	0.037409	1.440511	0.684464	0.063118	143.457138	13.228839	Iron(2,0,0)
81.999375	0	1.174134	0.684464	0	160.622371	0	Iron(2,1,1)
Sample heated to 600°C							
2-theta (deg)	Esd (2-θ)	D (ang.)	FWHM (deg)	Esd (FWHM)	Crystallite size(ang.)	Esd (Crystallite size)	Phase name
44.34257	0.010868	2.041187	0.45969	0.009136	194.910228	3.87363	Iron(1,1,0)
64.661665	0.032192	1.440326	0.664532	0.095174	147.767465	21.163232	Iron(2,0,0)
82.085	0	1.173126	0.664532	0	165.547503	0	Iron(2,1,1)
Sample heated to 1300°C							
2-theta (deg)	Esd (2-θ)	D (ang.)	FWHM (deg)	Esd (FWHM)	Crystallite size(ang.)	Esd (Crystallite size)	Phase name
44.307633	0.012013	2.042716	0.44708	0.010732	200.382649	4.810318	Iron(1,1,0)
64.832375	0	1.436945	0.44708	0	219.84664	0	Iron(2,0,0)
81.99775	0	1.174153	0.44708	0	245.904128	0	Iron(2,1,1)

From the above **Table 5.1**, we can notice that there was a gradual increase in the Crystallite size of the sample at the Iron(2,0,0) phase as the temperature increased, previously when the sample was at room temperature and at 600° there was an

observation of crystallite size measurement at two peaks, whereas for the sample at 1300° the crystalline measurement was possible only at one peak. the D'S pacing [d(A)] was almost the same for the sample at respective temperatures, so only the formation of α -Ferrite can be noticed. This inference was made from the data of JCPDS (Joint Committee on Powder Diffraction Standards) of the (ICDD) International Centre for Diffraction Data. The FWHM (Full Width Half Maxima) of the sample at room temperature is higher than that of the samples at the higher temperature, this indicates that the sample at room temperature is less crystalline than the samples at high temperatures. The sample has more mechanical properties at room temperature. The sample at higher temperatures is more crystalline in nature when compared to that of the sample at room temperature.

5.2 TGA AND DTA

The following **Table 5.2** illustrates the melting points and weight % of respective elements, which makes it easy to notify the element which gets evaporated till the respective temperature. and this data is useful in inferring DTA and TGA analyses

Table 5.2 Melting Points of Elements

ELEMENT	% wt.	MELTING POINT	BOILING POINT
P	0.106	44.1°C (317.25K)	280.0°C (553.15K)
S	0.001	112.8°C (385.95K)	444.6°C (717.75K)
Al	0.018	660.37°C (933.52)	2467.0°C (2740.15K)
Cu	0.249	1083.0°C (1356.15K)	2567.0°C (2840.15K)
Mn	0.36	1245.0°C (1518.15K)	1962.0°C (2235.15K)

ELEMENT	% wt.	MELTING POINT	BOILING POINT
Si	0.31	1410.0°C (1683.15K)	2355.0°C (2628.15K)
Ni	0.197	1453.0°C (1726.15K)	2732.0°C (3005.15K)
Co	0.006	1495.0°C (1768.15K)	2870.0°C (3143.15K)
Fe	98.095	1535.0°C (1808.15K)	2750.0°C (3023.15K)
Ti	0.004	1660°C (1933.15K)	3287.0°C (3505.15K)
Cr	0.5	1857.0°C (2130.15K)	2672.0°C (2945.15K)
V	0.003	1890.0°C (2163.15K)	3380.0°C (3653.15K)
Mo	0.021	2617.0°C (2890.15K)	4612.0°C (4885.15K)
W	0.01	3410.0°C (3683.15K)	5660.0°C (5933.15K)
C	0.12	3500.0°C (3773.15K)	4827.0°C (5100.15K)

The following is the graph of TGA analysis of the Fire Resistant Structural Steel of the 0.8 mm thick sample, taken till 1000°C.

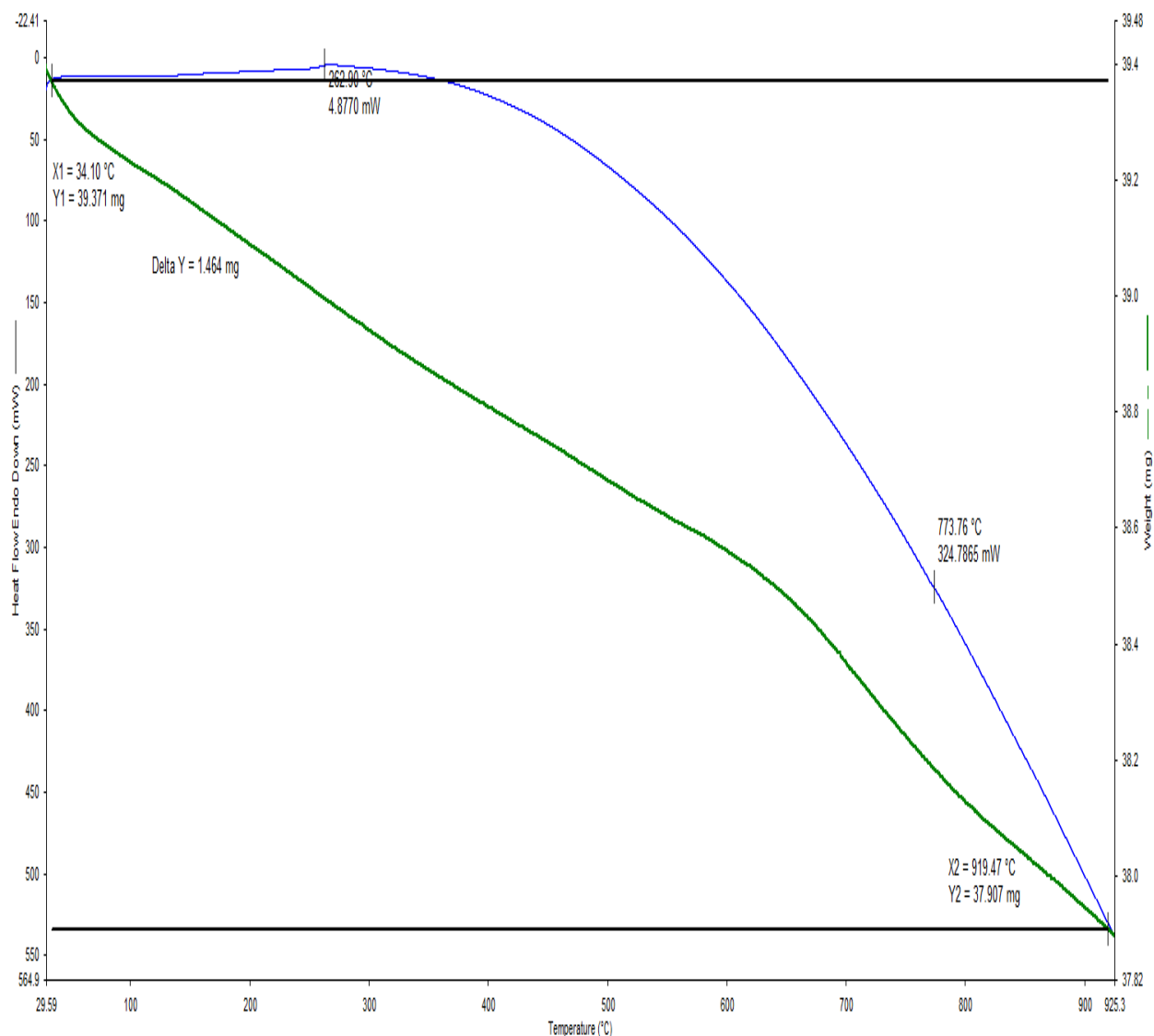


Figure 5.4 TGA and DTA graph up to 1000°C

From the above-given graph in the **Figure 5.4**, we can infer that there is a mass loss in the material when it is subjected to temperature up to 1000°C. There is a gradual decrease in the mass, which is because of the elements present in the material getting vaporized when they reach their melting point, as the temperature is maintained up to 1000°C, the elements having their melting point below this get vaporized. By the end of 1000°C, Phosphorus, Sulphur, Aluminum which comprises wt.% as 0.106%, 0.001%, 0.018% respectively are vaporized from the material. The weight loss of 1.464 grams from the total weight of the samples when it was subjected till 1000°C. And the DTA graph indicated that when the sample gets exposed to 1000°C, the

reaction is endothermic and has taken place with an increase in heat flow of 319.9095mW.

The following **Figure 5.5** is the combination of TGA and DSC graphs that resulted from the respective tests on Fire Resistant Structural Steel up to the temperature range of 1400°C.

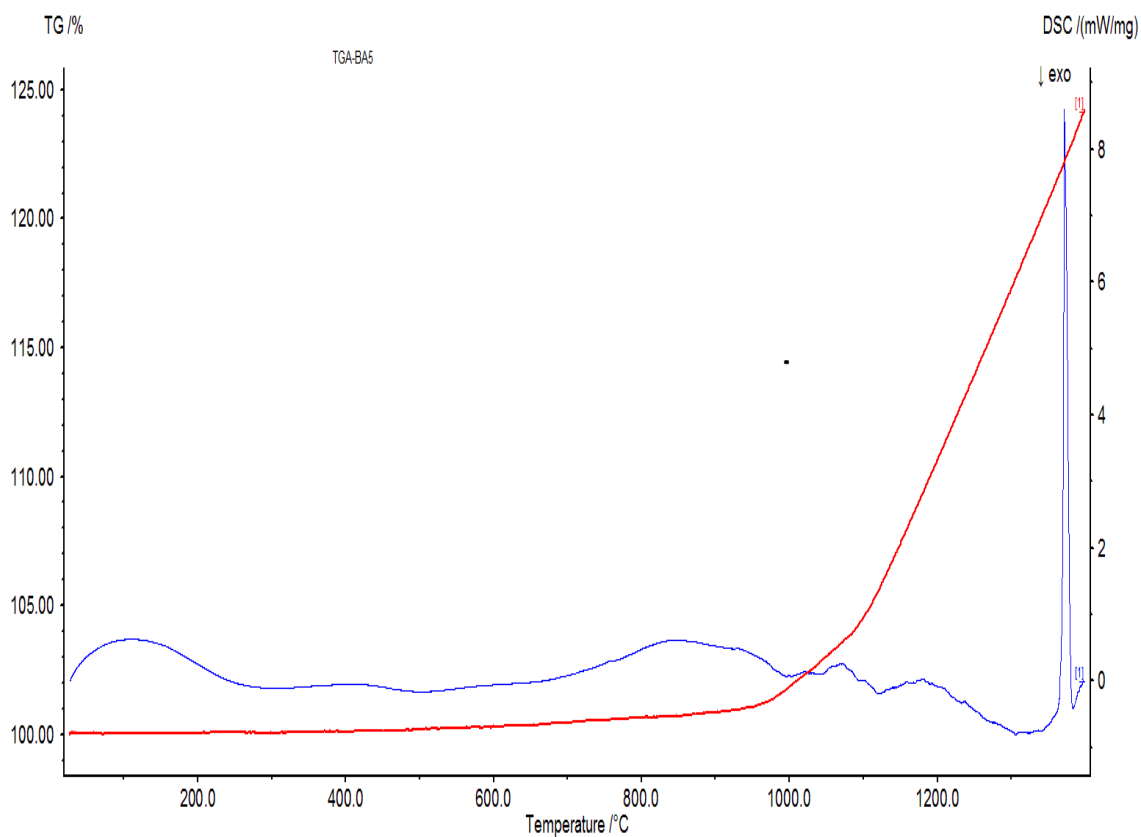


Figure 5.5 TGA graph till 1400°C

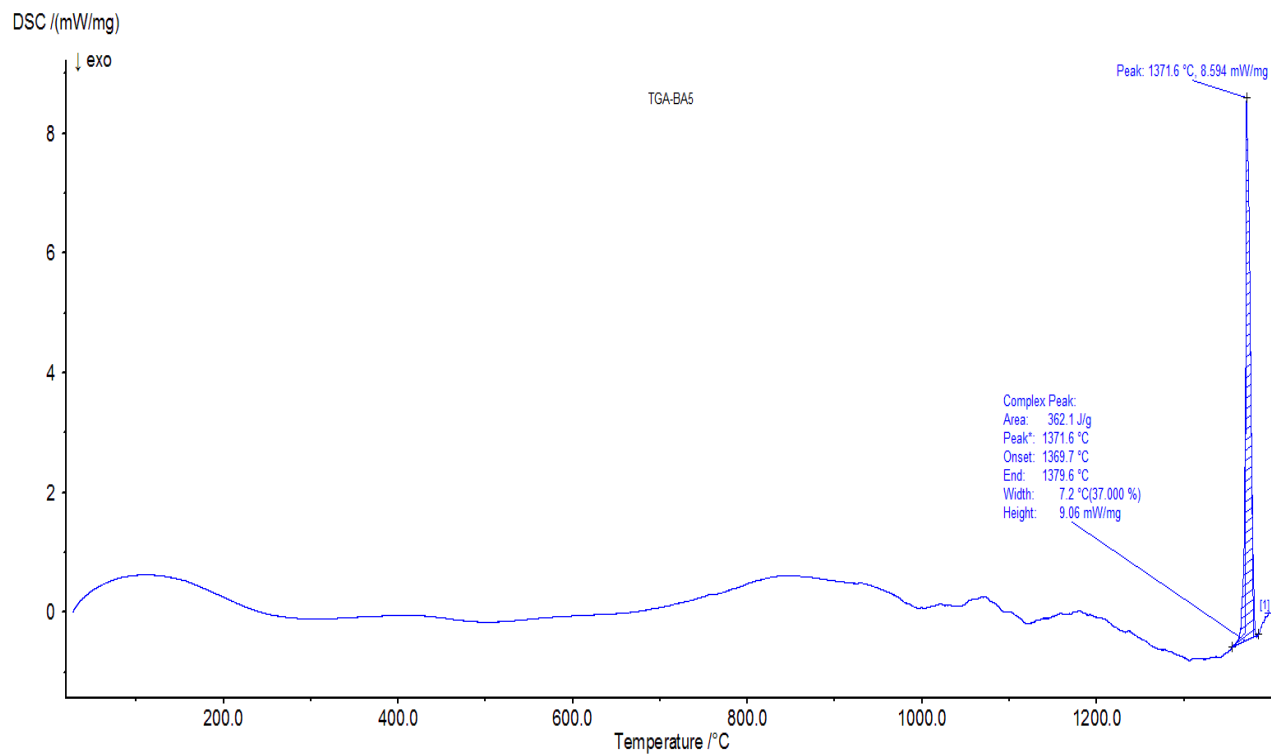


Figure 5.6 DSC graph till 1400°

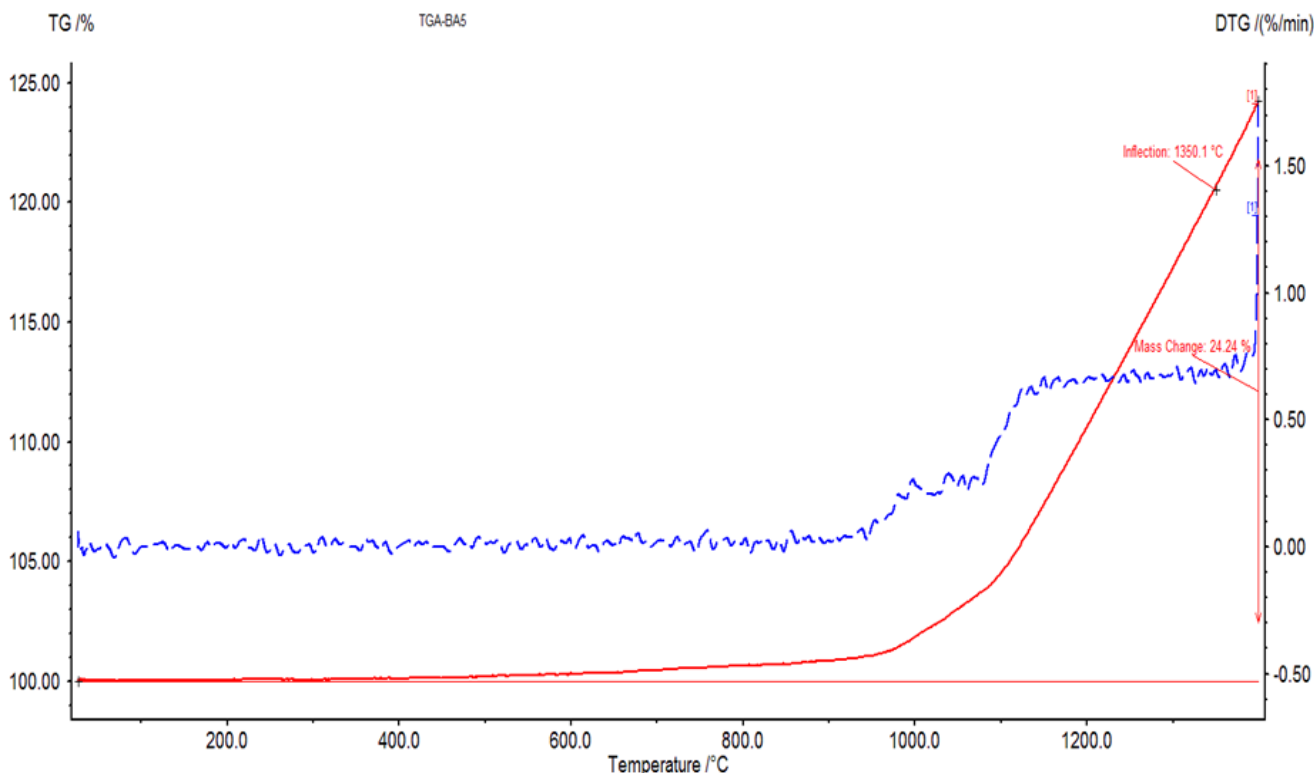


Figure 5.7 TGA and DTA graph

From the above given graphs in the **Figure 5.6** and **Figure 5.7**, we can infer that till the temperature close to 1370°C there are deflection in the TGA curve where the mass is seen constant but after 1370°C there is a huge gain of mass, this gives us the information that a chemical reaction occurred which led to the gain in mass of the material, and even from the DSC curve we can infer that endothermic reaction has taken place at 1371.6°C. The major gain of weight from the sample occurred after 1370°C, when the sample was subjected to 1400°C, and the weight gain percentage is 24.24% of the total weight of the sample, this is because of the reaction formed by the evaporated elements. The elements which got evaporated due to their lower melting point did not undergo any reaction until it was subjected to 1000°C, but when it was subjected to 1400°C, due to the endothermic reactions formation which was inferred from DTA graph, the sample has gained weight after 1370°C. The reaction formation might be due to the evaporated elements, which are subjective to the atmospheric gases at high temperatures, forming an endothermic reaction.

5.3 Results of welded Fire Resistant Structural steel

5.3.1 XRD test

XRD test was carried out on all the five samples using Rigaku, Japan with Cu K α with the radiation ranging from 20–80° with the step size of 0.02°.and the following graphs have obtained as shown in the **Figure 5.8**

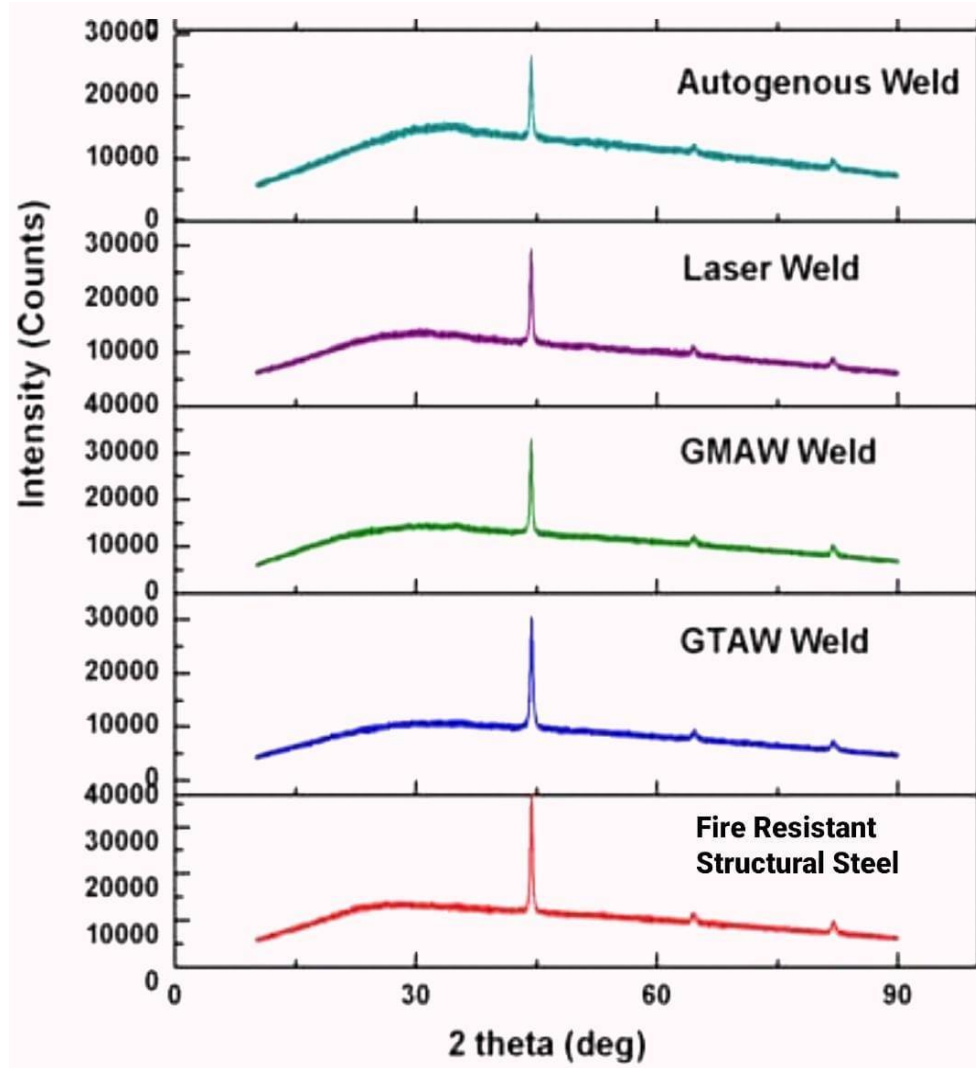


Figure 5.8 XRD Graphs of welded Fire Resistant Structural Steel

5.3.2 XRD results of Base metal (Fire Resistant Structural Steel)

The base metal sample was made to go through the XRD test, and the following observations were noted down.

As seen in the figure, the XRD pattern of Fire Resistant Structural steel Fe is associated with a cubic structure (JCPDS Card No: 089-7194) indexed. The Fe is pure and has a variety of constituent phases. The diffracted peaks of the Fe compound are found at 44.27° , 64.58° , and 81.95° , which corresponds to crystallographic diffraction peaks of (1,1,0), (2,0,0), and (2,1,1), respectively. The maximum intensity of the peak is observed at 44.27° , and its corresponding phase is Iron (1,1,0). The grain size of the 30 microns. The corresponding d spacing values [d(ang.)] are 2.044433, 1.441863, and 1.17477. The corresponding Crystallite size(ang.) values are 270.749752, 198.938328, and 222.734676. The corresponding Full-width Half Maxima (FWHM) values are 0.330839, 0.493391 and 0.493391.

5.3.3 XRD results of GTAW welded Fire Resistant Structural Steel

The GTAW welded sample was made to go through the XRD test, and the following observations were noted down.

As seen in the figure, the XRD pattern of GTAW welded sample Fe is associated with a cubic structure (JCPDS Card No: 089-7194) indexed. The Fe is pure and has a variety of constituent phases. The diffracted peaks of the Fe compound are found at 44.297399° , 64.499549° , and 82.00° , which corresponds to crystallographic diffraction peaks of (1,1,0), (2,0,0), and (2,1,1), respectively. The maximum intensity of the peak is observed at 44.297399° , and its corresponding phase is Iron (1,1,0). The grain size of the 50 microns. The corresponding d spacing values [d(ang.)] are 2.043164, 1.443554, and 1.174127. The corresponding Crystallite size(ang.) values are 231.88163, 206.98133, and 231.943927. The corresponding Full-width Half Maxima (FWHM) values are 0.386334, 0.473997, and 0.473997.

5.3.4 XRD results of GMAW welded Fire Resistant Structural Steel

The GMAW welded sample was made to go through the XRD test and the following observations were noted down.

As seen in the figure, the XRD pattern of GMAW welded sample Fe is associated with a cubic structure (JCPDS Card No: 089-7194) indexed. The Fe is pure and has a variety of constituent phases. The diffracted peaks of the Fe compound are found at

44.24⁰, 64.57⁰, and 81.92⁰, which corresponds to crystallographic diffraction peaks of (1,1,0), (2,0,0), and (2,1,1), respectively. The maximum intensity of the peak is observed at 44.243251⁰, and its corresponding phase is Iron (1,1,0). The grain size of the 20 microns. The corresponding d spacing values [d(ang.)] are 2.045539, 1.442185, and 1.17505. The corresponding Crystallite size(ang.) values are 282.707234, 204.674037, and 229.135. The corresponding Full-width Half Maxima (FWHM) values are 0.316818, 0.479522, and 0.479522.

The following **Table 5.3** illustrates the XRD data of four different welds.

Table 5.3 XRD data of different welds

	2-theta(deg)	d(ang.)	FWHM (deg)	Crystallite size(ang.)	Phase name
Fire Resistant Structural Steel	44.268434	2.044433	0.330839	270.749752	Iron(1,1,0)
	64.584371	1.441863	0.493391	198.938328	Iron(2,0,0)
	81.9455	1.17477	0.493391	222.734676	Iron(2,1,1)
GTAW Weld	44.297399	2.043164	0.386334	231.88163	Iron(1,1,0)
	64.499549	1.443554	0.473997	206.98133	Iron(2,0,0)
	82	1.174127	0.473997	231.943927	Iron(2,1,1)
GMAW Weld	44.243251	2.045539	0.316818	282.707234	Iron(1,1,0)
	64.568184	1.442185	0.479522	204.674037	Iron(2,0,0)

	81.9215	1.17505	0.479522	229.135	Iron(2,1,1)
LBW Weld	44.236682	2.045827	0.316483	282.999062	Iron(1,1,0)
	64.485193	1.443841	0.495556	197.960914	Iron(2,0,0)
	81.89725	1.17534	0.495556	221.680453	Iron(2,1,1)
AGTAW Weld	44.257644	2.044907	0.327892	273.172556	Iron(1,1,0)
	64.535308	1.44284	0.387509	253.22758	Iron(2,0,0)
	81.8605	1.175774	0.387509	283.412028	Iron(2,1,1)

5.3.5 XRD results of LBW welded Fire Resistant Structural Steel

The LBW welded sample was made to go through the XRD test and the following observations were noted down.

As seen in the figure, the XRD pattern of LBW welded sample Fe is associated with a cubic structure (JCPDS Card No: 089-7194) indexed. The Fe is pure and has a variety of constituent phases. The diffracted peaks of the Fe compound are found at 44.24° , 64.48° , and 81.90° , which corresponds to crystallographic diffraction peaks of (1,1,0), (2,0,0), and (2,1,1), respectively. The maximum intensity of the peak is observed at 44.236682° , and its corresponding phase is Iron (1,1,0). The grain size of the 08 microns. The corresponding d spacing values [d(ang.)] are 2.045827, 1.443841, and 1.17534. The corresponding Crystallite size(ang.) values are 282.999062, 197.960914, and 221.680453. The corresponding Full-width Half Maxima (FWHM) values are 0.316483, 0.495556, and 0.495556.

5.3.6 XRD results of AGTAW Welded Specimen

The AGTAW Welded sample was made to go through the XRD test, and the following observations were noted down. As seen in the figure, the XRD pattern of AGTAW Welded sample Fe is associated with a cubic structure (JCPDS Card No: 089-7194)

indexed. The Fe is pure and has a variety of constituent phases. The diffracted peaks of the Fe compound are found at 44.26° , 64.54° , and 81.86° , which corresponds to crystallographic diffraction peaks of (1,1,0), (2,0,0), and (2,1,1), respectively. The maximum intensity of the peak is observed at 44.257644° , and its corresponding phase is Iron (1,1,0). The grain size of the 50 microns. The corresponding d spacing values [d(ang.)] are 2.044907, 1.44284, and 1.175774. The corresponding Crystallite size(ang.) values are 273.172556, 253.22758, and 283.412028. The corresponding Full-width Half Maxima (FWHM) values are 0.327892, 0.387509, and 0.387509.

5.3.7 Discussion of welded samples

The cubic structure (JCPDS Card No: 089-7194) is indexed with the XRD pattern of α -Fe. Similarly, the XRD patterns of C, Cr, and Ni correspond to cubic structure (JCPDS Card No: 089-7194, 089-7194, and 089-7194) indices. As shown in the figure, the peak intensity of LBW welded sample is recognizably increased, this might be because of the presence of C, Cr, and Ni which is not even 0.4%, but they are not recorded on the diffracted peaks as the XRD instrument selects the elements which are more than 5%.

The following **Figure 5.9** illustrates the FWHM, d (ang.) and the 2θ values of different welded Fire Resistant Structural Steel at their highest intensity peaks:

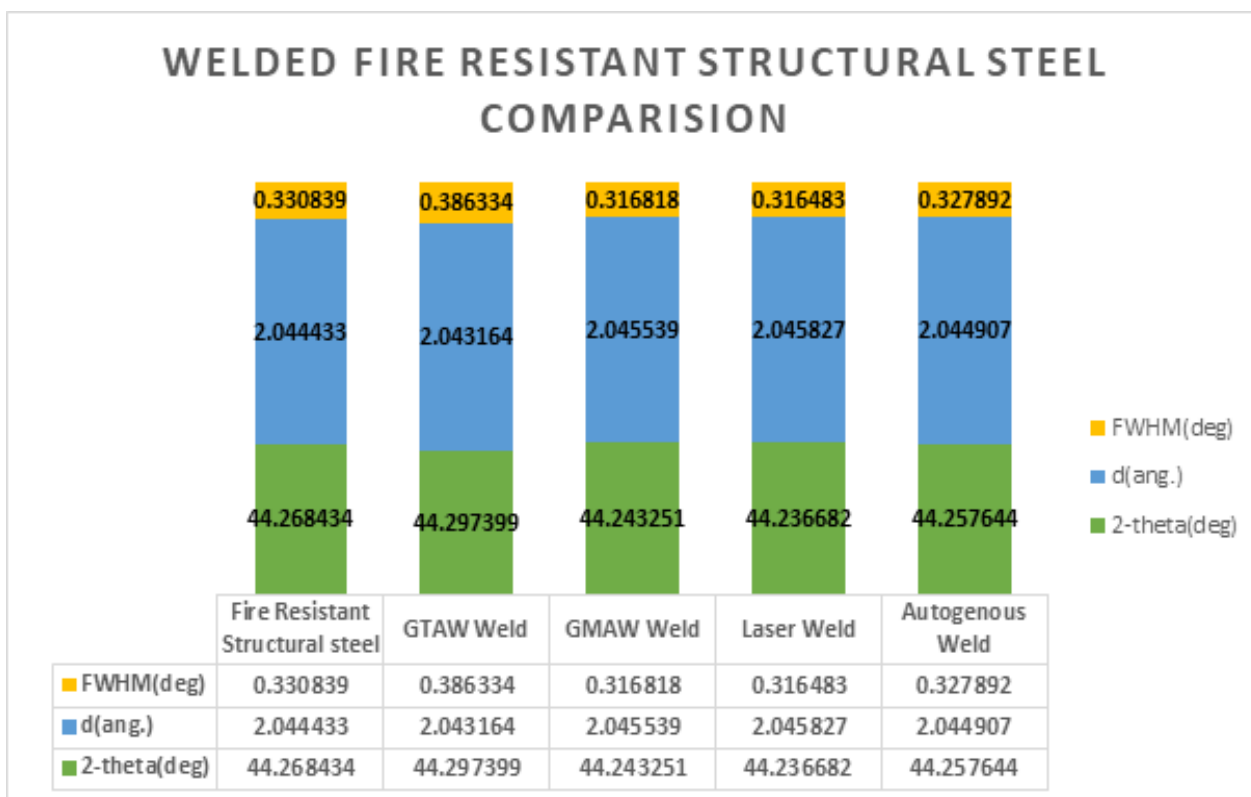


Figure 5.9 Welded XRD data comparison.

The following **Table 5.4** illustrates the 2-theta(deg), d(ang.),FWHM(deg) and Crystallite size (ang.)Major Intensity XRD Peak of welded and base metal:

Table 5.4 XRD data at Maximum Intensity peak

Type of Weld	2-theta (deg)	d (ang.)	FWHM (deg)	Crystallite size (ang.)
Fire Resistant Structural steel	44.268434	2.044433	0.330839	270.749752
GTAW Weld	44.297399	2.043164	0.386334	231.88163
GMAW Weld	44.243251	2.045539	0.316818	282.707234
LBW Weld	44.236682	2.045827	0.316483	282.999062
AGTAW Weld	44.257644	2.044907	0.327892	273.172556

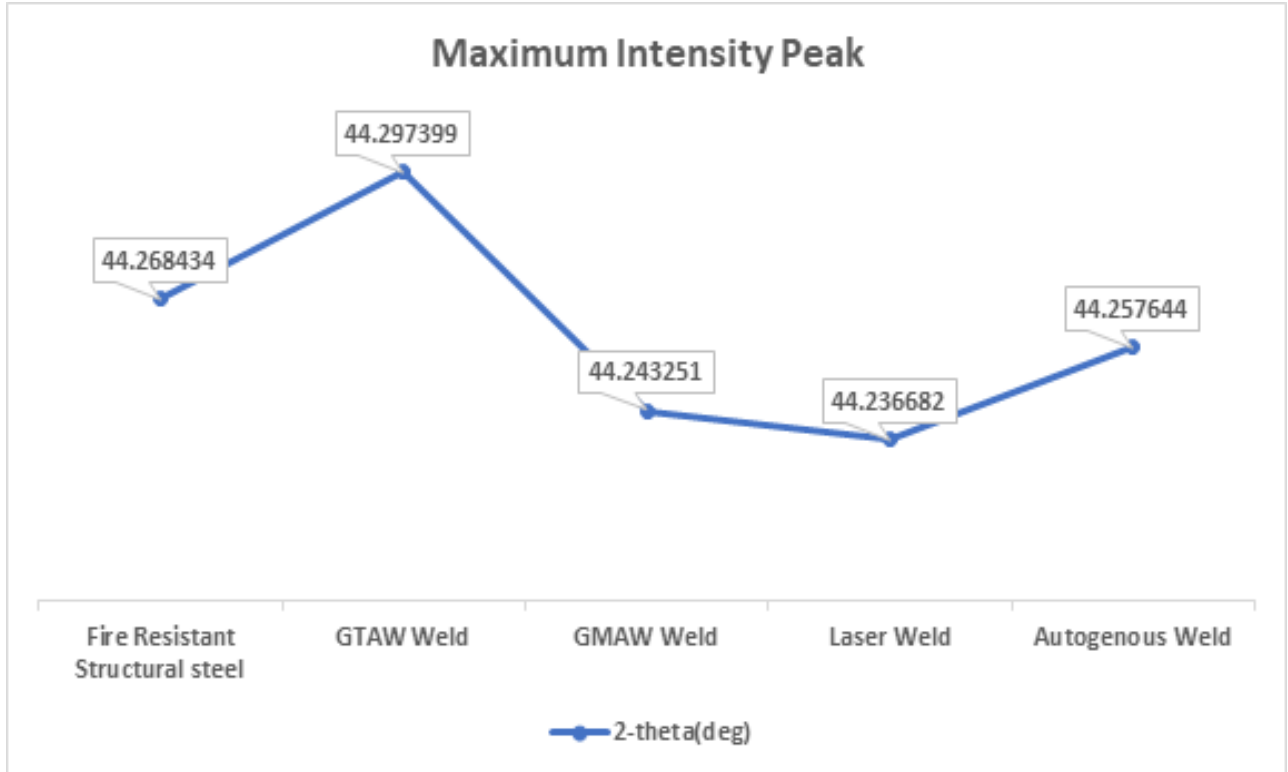


Figure 5.10 Welded 2-Theta(deg) graph

From the **Figure 5.10** and **Figure 5.11** the following observation are made. The intensity of the peak has also increased drastically for the GMAW, whereas for the GTAW and AGTAW the intensity has decreased. This may be attributed to C and Cr segregation from the grain boundary to the grain. Residual compressive stress is present in welded samples if the lattice constant value is greater than the normal JCPDS (Joint Committee on Powder Diffraction Standards) value. The d value of the base Fire Resistant Structural steel plate 2.0444 d (ang.) is greater than the d value of standard JCPDS 06-0696 (-Fe) 2.0268 d (ang.), indicating that the Fire Resistant Structural rolled steel plate is exposed to residual compressive stress. The case is similar to GTAW, GMAW, LBW weld, and AGTAW Weld, where the d value is more than base metal. This is because of the application of heat during welding.

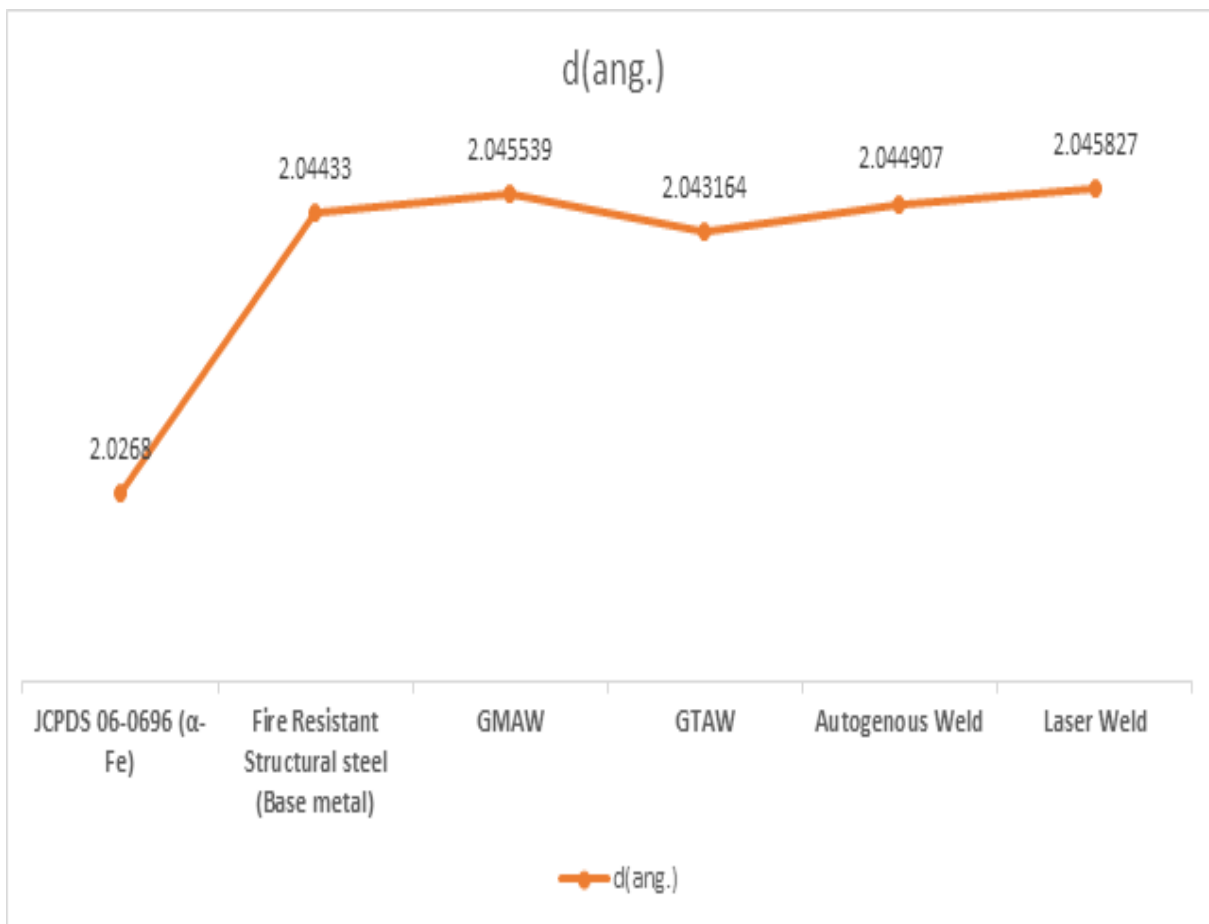


Figure 5.11 Welded samples d(ang.) comparison.

The LBW welded Fire Resistant Structural Steel has better mechanical properties than the base metal as the FWHM (Full Width Half Maxima) value of the LBW welded Fire Resistant Structural steel plate is more than that of base metal. As the Heat input for LBW welding is less, the FWHM value is more, whereas for the AGTAW the heat input is more which leads to the decrease in FWHM value and resulting in inferior mechanical properties when compared to LBW weld. The above data has been observed from the **Figure 5.12**

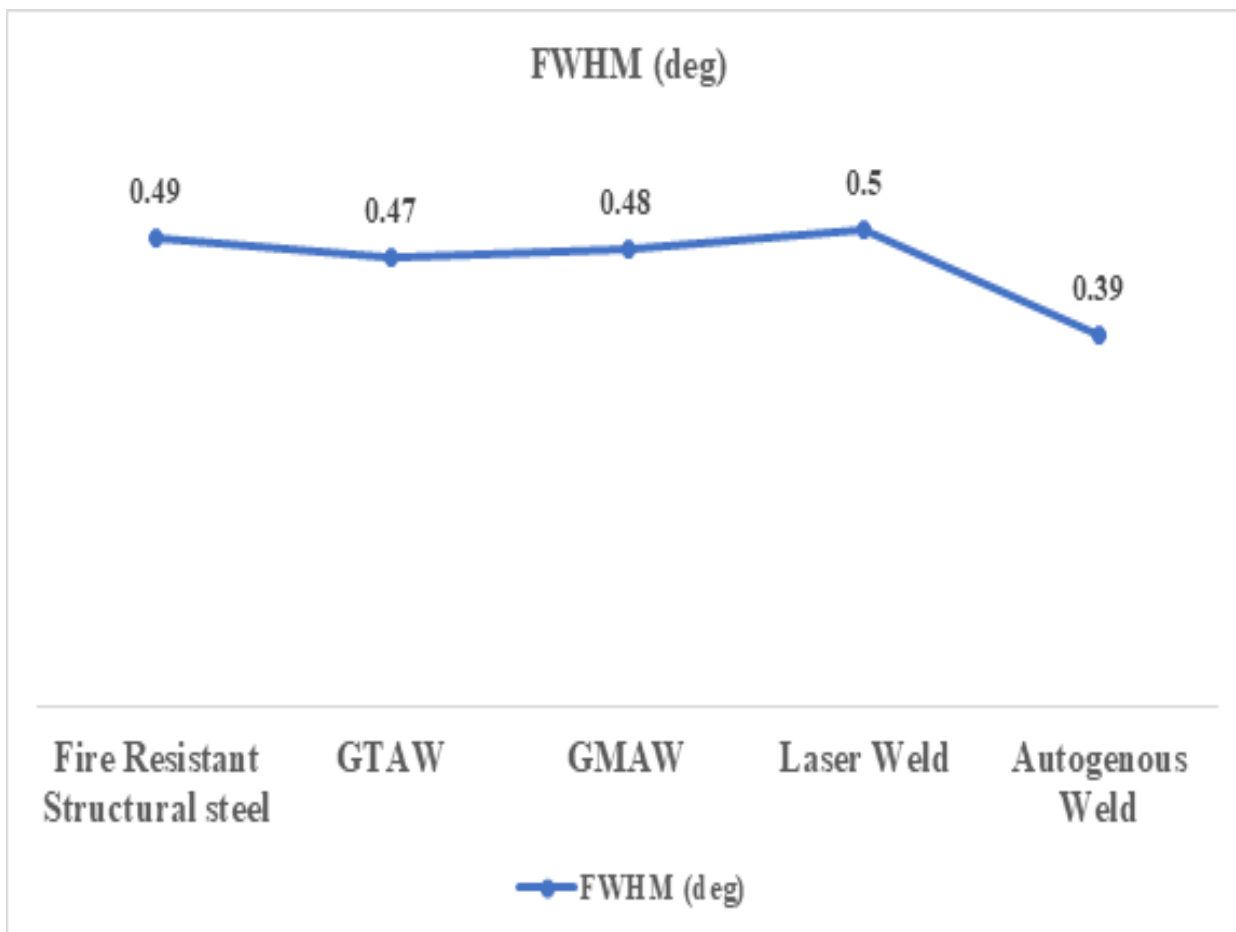


Figure 5.12 Welded FWHM value comparison.

The crystallite size of the Welded Fire Resistant Structural steel is more than the base metal except for GTAW, as the intensity of GTAW welded Fire Resistant Structural steel is higher, its periodicity is high, which resulted in the decrease of crystallite size. broadening of peak results in a decrease of crystallite size. Hence the crystalline size of the LBW-welded sample is the highest among all the welded samples. This data can be inferred from **Figure 5.13**

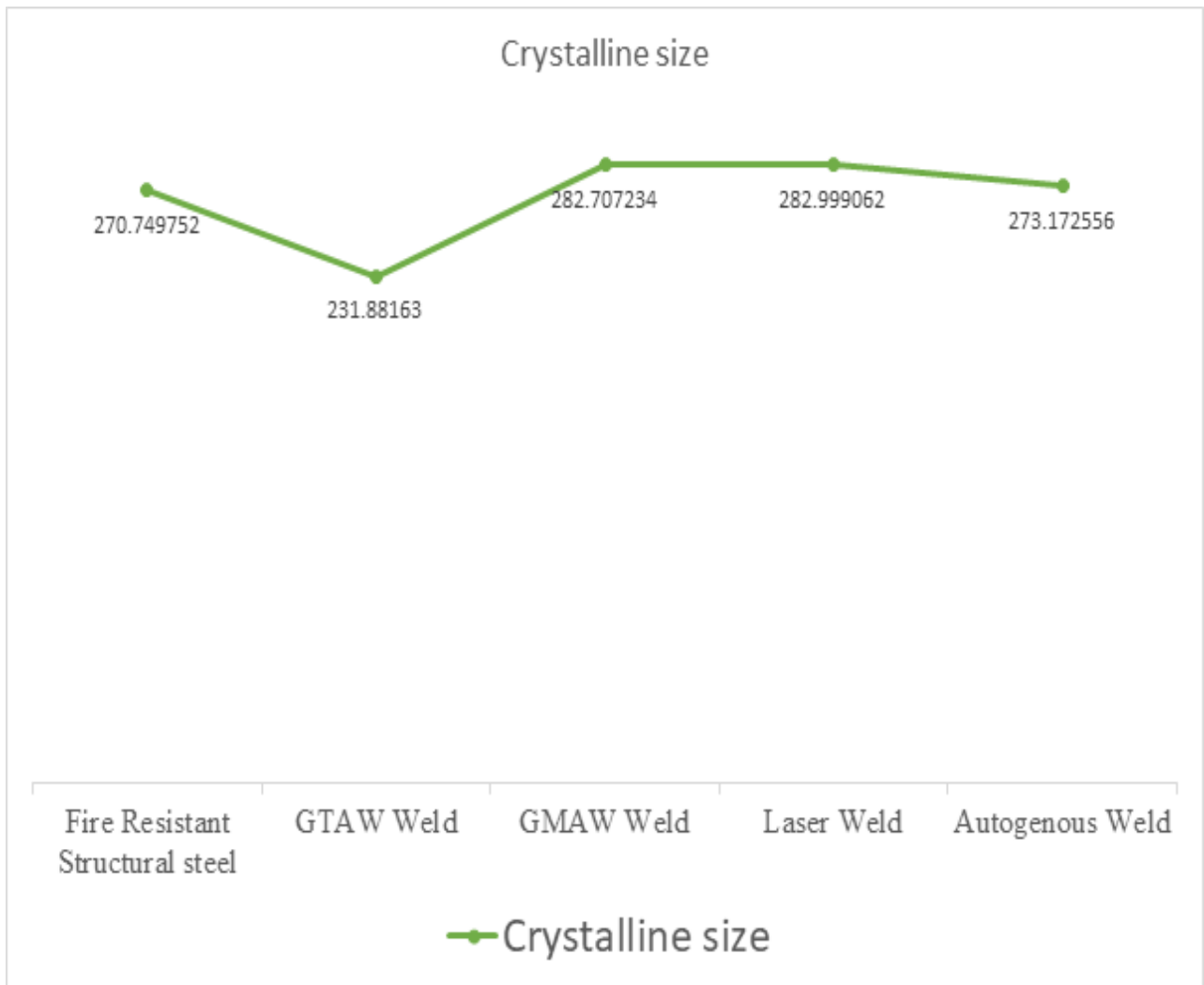


Figure 5.13 Welded Crystalline Size graph

The grain size is calculated in microns. The GTAW welded sample has the highest grain size among all the welds because it has the lowest d value among those welds, whereas the LBW-welded sample has the highest d value which resulted in the lowest grain size among all the welded samples. As the grain size decreases, the total area of the grain boundary increases, which leads to increase in the strength of the material, so the LBW welded sample is stronger than other welded Fire Resistant Structural steel samples and the base metal also. This data can be inferred from **Figure 5.14**

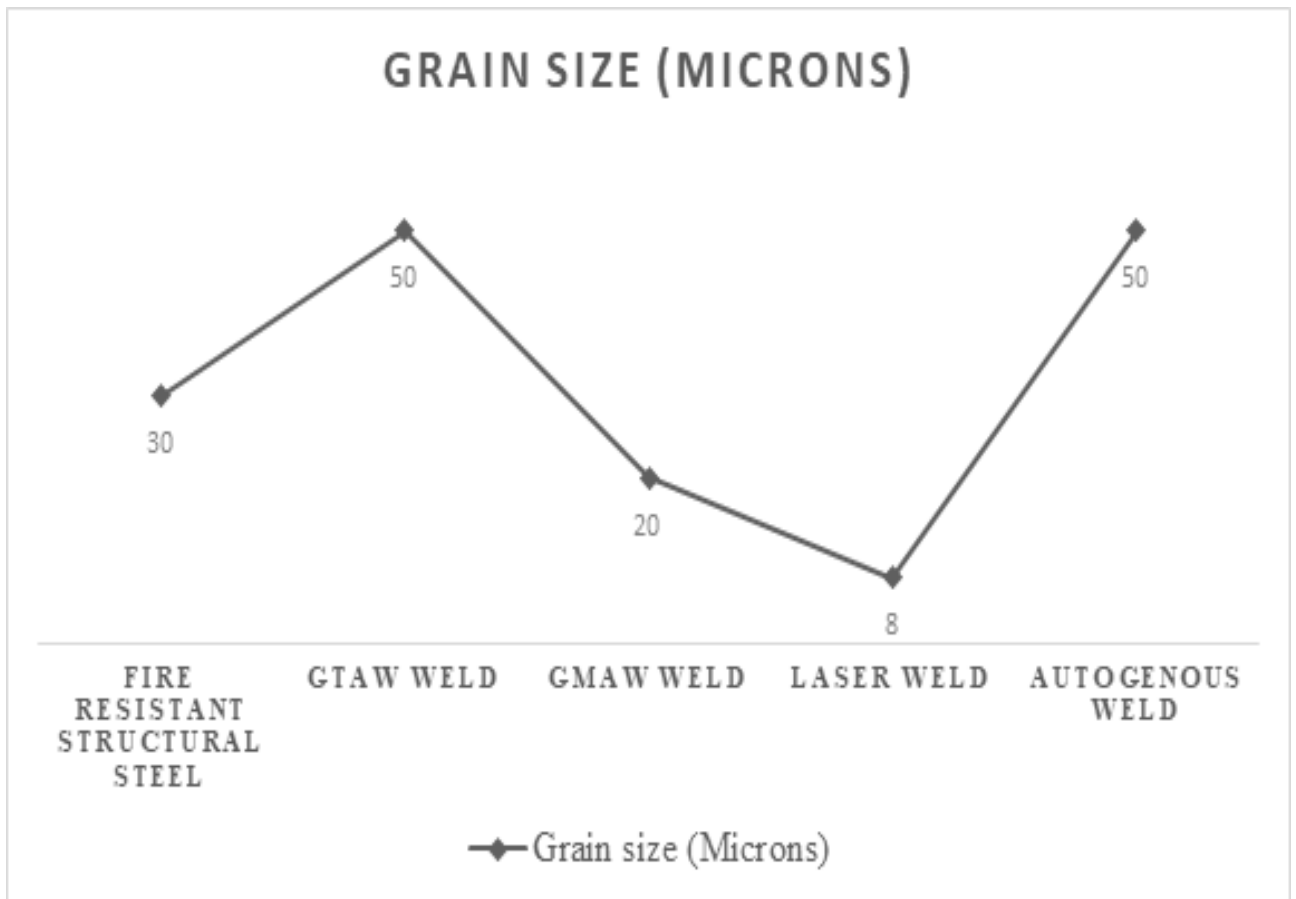


Figure 5.14 Welded Grainsize comparison graph.

CHAPTER 6

SUMMARY AND CONCLUSION

6.1 SUMMARY

- The HTXRD phases were the same at all the temperatures ranging from 600° to 1400°, and the phase was α -Ferrite.
- The crystalline size increased for the Iron (2,0,0) phase, with the increase in temperature.
- The D'S spacing [d(ang.)] was almost the same for the sample at all temperatures ranging from 600°C to 1400°C, which was nearly 2.041187 for the highest peak.
- The highest intensity peak was observed at 44.397358 for the sample at room temperature, almost the same resemblance was noticed with the sample at higher temperatures.
- The FWHM (Full Width Half Maxima) for the Fire Resistant Structural Steel at room temperature is higher than the samples at higher temperatures.
- The TGA graph indicated that there was a loss of 1.464 grams from the total weight of the samples when it was subjected till 1000°C.
- The major gain of weight from the sample occurred after 1370°C, when the sample was subjected to 1400°C, and the weight gain percentage is 24.24% of the total weight of the sample, this is because of the reaction formed by the evaporated elements.
- The weight loss was observed when the sample was treated till 1000°C, because of the evaporation of elements which have low melting points such as, Phosphorus, Sulphur, Aluminum which compromises wt.% as 0.106%, 0.001 %, 0.018%.
- The DTA graph indicated that when the sample gets exposed to 1000°C, the reaction is endothermic has taken place with increase in heat flow of 319.9095mW
- The DTA graph indicated that when the sample gets exposed to 1400°C, the

reaction taken place is endothermic, the peak observation was made at 1371.6°C.

- All the welded samples of Fire Resistant Structural Steel including the parent metal showed the phase of α -ferrite or pure Iron from the XRD analysis
- The highest peak intensity is observed in the GMAW welded sample among all the welded samples.
- The residual compressive stress is developed in the base metal and the welded samples as their d (ang.) value is 2.04433, which is higher than the JCPDS standard (2.0268). In welded samples the decline in d (ang.) value might be because of the heat input given while welding.
- As per FWHM values, LBW welding which has 0.5, is more suitable for Fire Resistant Structural steel (0.49) to enhance the parent metal's mechanical properties.
- As per the crystallite size the LBW welded Fire Resistant Structural steel has shown the highest value among all other welds with value of 282.999.
- The grain size of the LBW welded is 8 microns, which is the lowest, that leads to increase in the grain boundary and that strengthens the material. Hence LBW welded Fire Resistant Structural steel is the strongest of all welded Fire Resistant Structural steel and base metal.
- When the welded Fire Resistant Structural Steels and non-welded Fire Resistant Structural Steels are compared, LBW welded steel showed exceptional properties, making the LBW weld more suitable for Fire Resistant Structural Steel.

6.2 CONCLUSION

Based on the discussions made from the obtained results, the metallurgical behavior of Fire Resistant Structural Steel at elevated temperatures is known. From the weldments of the Fire Resistant Structural Steel, a meliorate weld with better metallurgical properties which is suitable for the steel among all the welds is suggested.

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APPENDIX

Plagiarism Report

Metallurgical Characterization and High-Temperature XRD (HTXRD) Investigation of Fire Resistance Structural Steel

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XRD Investigation of GTAW, GMAW, LBW and AGTAW Corten A588 grade steel weldments

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Abstract:	Corten A588 grade Steel is a weathering with low carbon content. It is a high strength low alloy steel with exceptional corrosion resistant property. When the Corten A588 steel is welded, due to high temperatures, few elements in the base metal get vaporized, thereby changing the crystalline nature and phases of the steel as the elements possess low melting points. The effects of GTAW (Gas Tungsten Arc Welding), GMAW (Gas Metal Arc Welding), LBW (Laser Beam Welding) and AGTAW (Autogenous Gas Tungsten Arc Welding) based on the heat input and various other parameters is compared. In this research GTAW, GMAW, LBW and AGTAW Corten A588 grade steel weldments were prepared and XRD test is performed to investigate the crystal structure. The d (ang.), phase identification, FWHM value, crystalline size is studied and a comparative analysis of all the weld which mellorates the mechanical and metallurgical nature of the Corten A588 is to be considered.

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To: <deepakcage@gmail.com>

Dear J R DEEPAK James Raj

We are glad to inform you that your paper titled " Laser Welding of ASTM Corten A588 grade steel - A Case Study" (Paper Id: --- MATPR-D-21-04209 -----) has been **PROVISIONALLY ACCEPTED WITH REVISIONS** for online presentation and publication in the " Materials today Proceedings " through FRESM'21 to be held online organized by Department of Mechanical Engineering, CMR Institute of Technology, Bengaluru, India & Core-Facility Centre for Photochemistry and Nanomaterials Gyeongsang National University, South Korea during **April 23-24, 2021**.

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