STUDY ON EFFECT OF HEAT TREATMENT ON THE MICROSTRUCTRUAL EVOLUATION & MECHANICAL PROPERTIES OF S2205 (DUPLEX STAINLESS STEEL)

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Abstract- Duplex stainless steels offer better corrosion resistance and mechanical properties than Austenitic stainless steels because of its unique microstructure which consists of ferrite and austenite phase distributed in equal proportions. From literature it is known that duplex stainless steels are stable only below 300ºC and above this temperature, the duplex steels will undergo micro structural change and in turn will affect the mechanical properties. There are many intermetallic phases found in duplex steel but the sigma phase is very important since its formation is around 700 to 950ºC. The sigma phase occurs is rich in Chromium and Molybdenum content. The peak temperature is around 900ºC were ferrite will be decomposed into sigma phase and it may take as little as two minutes in super duplex alloys. In this regard our study was aimed to study the influence effect of heat treatment temperature on the microstructure and mechanical properties of S2205 duplex stainless steel. In our investigation the solutionised samples were heat treated at various temperatures and all the samples were evaluated. The results showed that maximum amount sigma phase was induced at 900°C for 60 minute which was validated by tensile and microstructures results.

Index Terms—duplex stainless steel, heat treatment, micro structural analysis.scanaing electron microscope , x-ray diffraction, Vickers micro hardness,

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1 INTRODUCTION

Steels are iron alloys with varying carbon content from 0.002% to 2.1% by weight. Even in a narrow range of concentration of mixture of carbon and iron that make a steel, a number of different metallurgical structure, with very different properties can form. They are classified further based on their composition such as: Carbon steel, Alloy steel, Stainless steel, Tool steel (AISI). Stainless steels are iron alloys containing at least 11% chromium by weight. Due to the passive film of chromium oxide which forms on the surface of the steel, they demonstrate superior corrosion resistance compared to other steels. However, in order for a stainless steel to withhold its "stainless-ness" in pugnacious chemical environments, larger amounts of chromium needs to be added to the alloy (Lipoid and Kopeck (2005)). In order to provide superior resistance to different forms of corrosion other alloying elements such as nickel, molybdenum, manganese, nitrogen etc. are also need to be added.There are some alloying elements which is also added to enhance the mechanical properties and weld ability without adjusting on the need of corrosion resistance (Ki Leak Lai et al. (2012)). On their basis of microstructure, Stainless steels can be distinguished into five groups such as: Ferritic, Austenitic, Martens tic, Duplex and Precipitation Hardening stainless steels (Davis 1994).

Duplex stainless steels (DSS) contains a two phase microstructure which are ferrite and austenite in nearly even handed proportions and it offers better corrosion resistance and mechanical properties in comparison to single phase stainless steels (Kazior et al. (2004)). It provides excellent resistance against pitting corrosion and stress corrosion cracking even in the chloride environment (Singh Raman and Siew (2010)). DSS can be beyond classified mainly into three groups based on their pitting resistance equivalent numbers (PREN). When the alloys are having a PREN feebly higher than that of austenitic stainless steel grades and contain up to 20 wt% Chromium and no molybdenum then it is called as "Lean Duplex". When the, PREN is lying between 33 and 36, and it is consisting of the alloys with around 22 wt% Chromium and 3 wt% Molybdenum then it is called as Standard Duplex. Then it comes to "Super Duplex" alloys with more than 25 wt% Chromium, 3.5 wt% Molybdenum and 0.2 - 0.3 wt% Nitrogen, with a PREN greater than 40. Recently, special grades called "Hyper Duplex" stainless steels with much higher chromium and nitrogen levels have also been developed (Alvarez-Arms and Degallaix-Moreuil (2009)). Duplex stainless steels have various industrial applications which involves chemical and petrochemical, oil and gas, pulp and paper, power generation, hydrometallurgy, marine transportation, construction industry etc. (Gunn (1997))..In application of DSS it is having one major limiting factor which is they are vulnerable to thermal embrittlement when they are exposed to temperature range from 280^0C to 525^0C. This form of Embrittlement is popularly known as "475^0C embrittlement" since the rate of embrittlement within this temperature range is maximum at 475^0C (Chung (1992)).

Studies conducted on ferritic and duplex stainless steels have shown that the changes in microstructure and mechanical properties associated with the 475^0C embrittlement can be undone by subjecting the material to a short term “reversion” heat treatment within the range of 550-600^0C (Kenos (1992). However, when cast austenitic stainless steels are exposed to the 550 ºc or higher for a long time, various types of detrimental intermetallic phases, such as sigma (σ) phase, Chi (χ) phase, R phase, and M23C6 carbides could be formed in the ferrite phase or at ferrite phase boundaries. The sigma (σ) phase occurs in range between 650^0C to 1000^0C.Sigma phase is rich with Chromium and Molybdenum component.

At the peak temperature of around 900^0C ferrite decomposition to sigma may take as little as two minutes in super duplex alloys. From past experiments results shown that Chromium, Silicon, Manganese and Molybdenum encourage sigma formation in duplex stainless steel. Sigma phase cause decrement in impact resistance and corrosion resistance In this regard, the Scope of present work was aimed to develop an economic approach to extend the service life of long term working duplex stainless steel components which are susceptible for decrement in corrosion resistance and impact resistance. The purpose of this investigation is, to study the mechanical behavior of a duplex stainless steel, when subjected to heat treatment of different durations.

2 EXPERIMENTAL

2.1 MATERIAL

Rods with 20mm diameter made of Duplex stainless steelsS2205have been used for the experiment. The DSS S2205 rod is composed of chemical elements as follows:-

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Table 1: Chemical composition of the specimen (wt %)

C	0.02
Si	0.34
Cr	25.34
Mn	0.64
N	0.27
Ni	6.77
Mo	3.45
Cu	0.51
W	0.72

2.2 HEAT TREATMENT

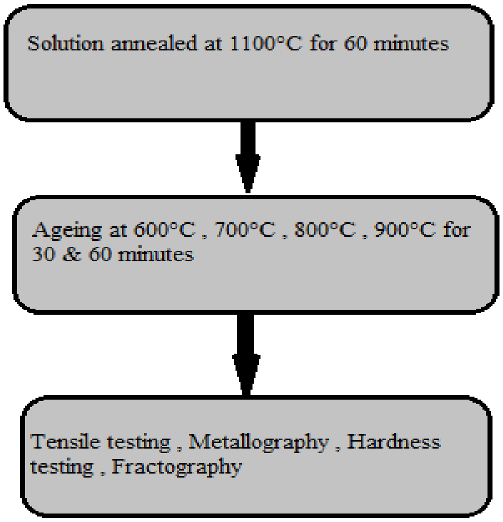

Fig1 This Flow diagram represents the thermal ageing and heat treatment processes followed in this study

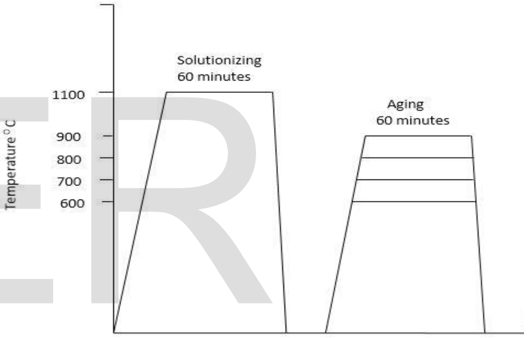

Figure 2 Heat treatment cycle

The specimen was heated to 1100ºC and will be kept in this condition for 60 minutes followed by rapid decrement in temperature through water quenching. Afterwards those samples were heated at temperature of 600°C, 700°C, 800°C, and 900°C for 30 & 60 minutes later they were quenched by water.

2.2 MICROSTRUCTURAL ANALYSIS

Through literature it is evident that change in Microstructure can impose very critical effect on the mechanical behavior of material, so that the necessary microstructure observation and mechanical test were used to analyze microstructure and mechanical properties of specimens.

2. Scanning Electron Microscopy (SEM)

The microstructures of all product forms were also analyzed at higher magnifications in a Scanning electron microscope using the secondary electron (SE) and backscattered imaging modes.

2.4 X-ray Diffraction

To identify the phases present in the heat treated samples,

samples were subjected to X-ray diffraction studies. The samples were characterized by using X-ray diffractometer, (operated with Cu-Kα radiation at 30KV and 20mA). The 2θ range was selected between 30° to 100°. This range was selected because for our samples all the major intense peaks of the phases expected in the sample are present in this region. X-ray diffraction profile consisted of two peaks within this angular range namely the (111) peak of austenite and the (110) peak of ferrite. Through Direct comparison method as per suggested by Cullity (1974)the volume fraction of austenite and ferrite could be determined.By measuring the area under the peaks using the PeakAnalyzer operation in Origin 8.0 integrated intensity of the peaks was obtained.

2.5Tensile strength test:

One of the most fundamental tests for engineering is Tensile testing, and it provides valuable information about a material and its associated properties. These properties can be considered as a part for design and analysis of complex engineering structures, and for developing new materials which is design for their specified use.
Tensile properties of the specimens were measured in both annealed & aged conditions. Tensile strength, yield strength and ductility were determined at a strain rate 1mm/min using Shimadzu 100kN Universal Testing Machine.Through software the data was gathered, and loaded into Excel spreadsheet.A schematic diagram of tensile test specimen is as shown in the fig.

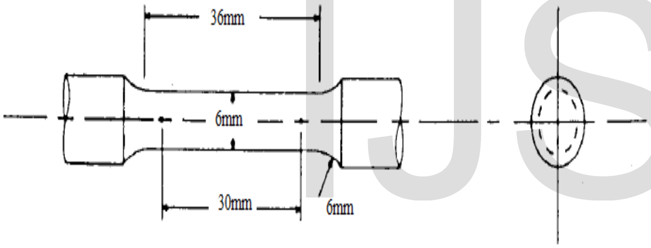

Fig 3 tensile test specimen

2.6 Fractography

The fracture surfaces of the tensile tested specimens under each heat treated condition, were examined in a scanning electron microscope to analyze the fracture features and attempt to relate the topography of the fracture surface to the mechanism of fracture. The approach to procedure was enough concerned to not damage fractured surface chemically and mechanically.

2.7 Vickers micro hardness

In general, hardness means resistivity to the physical change due to external forces, and for the metals hardness is a measurable unit which indicate their resistance to permanent or plastic deformation. The hardness of a metal or an alloy may be defined in one or more of the following ways like indentation hardness, rebound hardness, scratch hardness, cutting hardness, and abrasive hardness. To the base of various complex systems to analyze the hardness of various metals in industrious use one of above principals have played their crucial roles. These testing machines are widely used in the field of research and industry.

For sack of our experimental work,Vickers micro hardness was used.Zwick Vickers hardness tester is used to measure Vickers micro hardness values.Specimens were polished as for microscopic study and etched withCarpenters Stainless Steel Etchant. Specimens were solution annealed at 1100ºC for 60 minutes then aged at 600ºC, 700ºC, 800ºC, 900ºC for 30 minutes & 60 minutes.The micro-hardness values of ferrite and austenite phases were taken in ferrite phase and in austenite phase with a load of 10 g for 30 seconds.Readings were tabulated and Hardness profile was obtained.

3 RESULT AND DISCUSSION

3.1 Scanning Electron Microscopy (SEM)

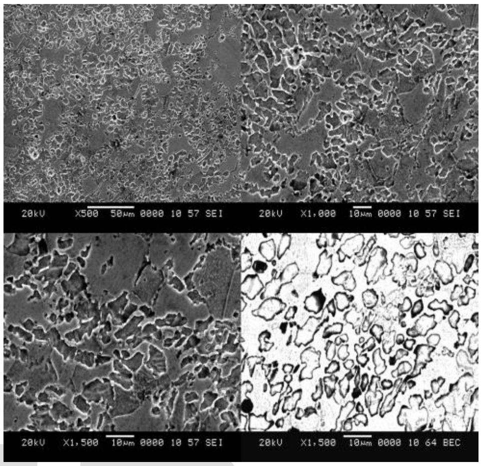

Fig 4 SEM image of as received SDSS

Figure 4-1 shows the SEM micrograph of the as-received S2205 DSS in the wrought form in which austenitic islands are embedded in the ferritic matrix with some undisclosed precipitates. Hence, in order to dissolve these harmful precipitates samples were subjected to solution heat treatment by heating it to 1100 ºC for 60 minutes. The solution heat treatment was also done to adjust the austenite and ferrite phase proportions and to eliminate the macro segregations which can be seen

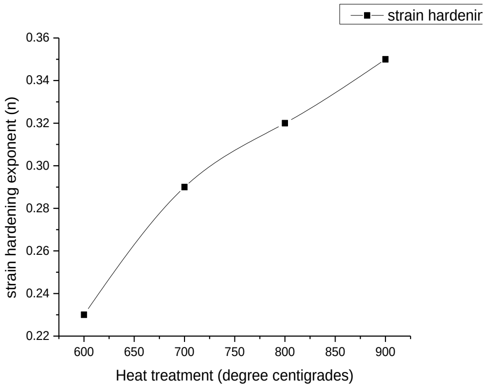

Fig 5 Variation of strain hardening exponent with Aging time

The yield strength of 563 MPa and the ultimate tensile strength of 767 MPa with 46% ductility was obtained in the solution heat treated condition. The fractured surface of solution heat treated tensile sample observed under SEM and is as shown in the Figure The fractured surface morphology was fully ductile with dimples and no inclusions were found throughout the fracture surface.

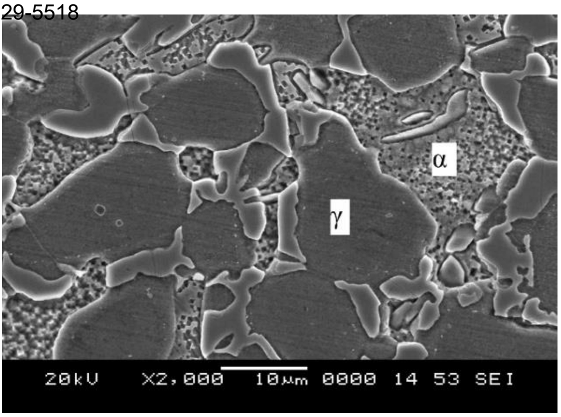

Fig 6 SEM images of the aged samples - 900°C for 60 minutes [magnification 2000X]

After solutionizing the specimen at 1100^0C temperature for 60 minute, the samples had been subjected to the ageing heat treatment process in order to study the ageing behaviors with respect to mechanical and micro structural characterization. From the figure 4-29 and figure 4-30 we can see that the both yield strength and the ultimate tensile strength was increasing with increase in the ageing temperature because of the formation of the sigma formation in the ferritic phase. Since the diffusion kinetics of the Molybdenum and the Chromium atoms rapidly increases the rate of formation of the sigma phase also increases with increase in the ageing temperature. This diffusion usually take place at boundary of ferrite-ferrite or ferrite-austenite it will occur only in ferrite composition as seen in the SEM images 4-6 to 4–25. As the Molybdenum and Chromium atoms diffuse to form sigma precipitate it will leave a void in the ferrite structure as both of them are ferrite stabilizer and it will cause the conversion of ferrite into the austenite, which finally affects tensile strength of specimen.

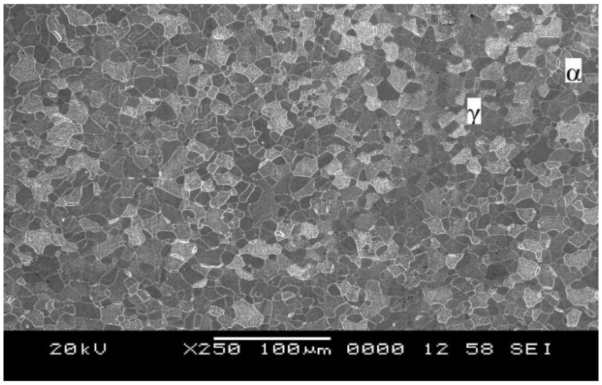

Figure 7 SEM image of the solutionized sample at magnification 250X

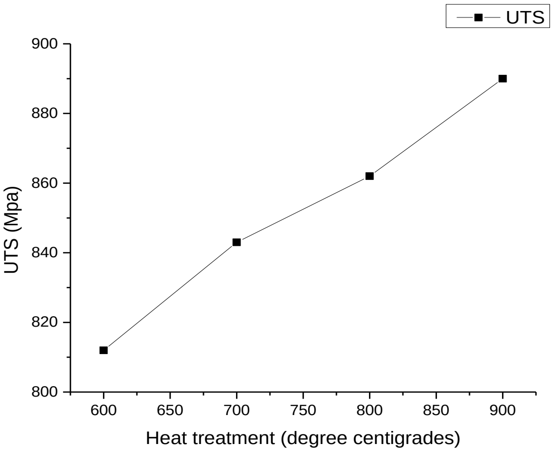

Fig 8 variation of UTS with aging time

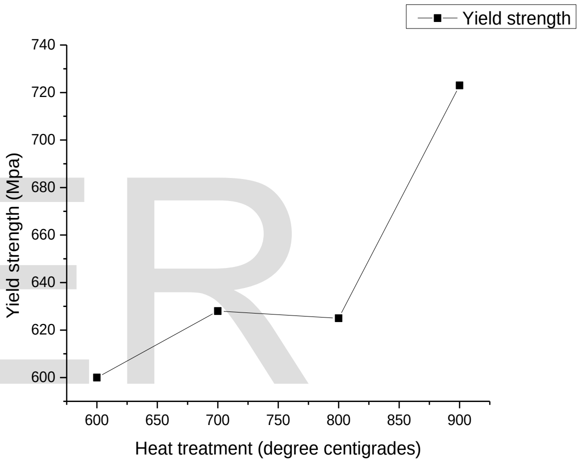

Fig 9 variation of Yield strength with aging time

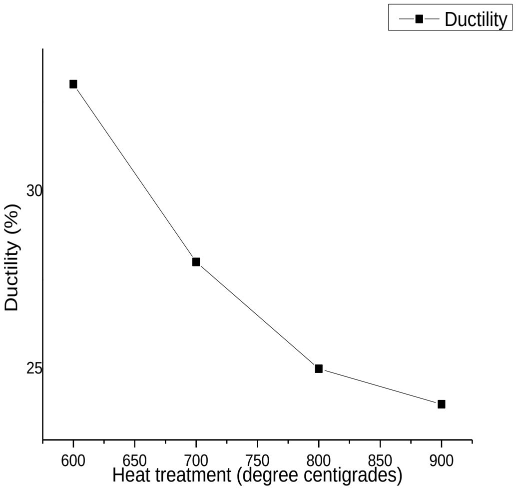

Figure 10 variation of ductility with aging time

3.2 Effect of Solutionizing heat treatment

The yield strength of 563 MPa and the ultimate tensile strength of 767 MPa with 46% ductility was obtained in the solution heat treated condition. The fractured surface of solution heat treated tensile sample observed under SEM and is as shown in the Figure 4.33. The fractured surface morphology was fully ductile with dimples and no inclusions were found throughout the fracture surface.

3.3 Effect of ageing on the microstructure

Duplex stainless steels exhibited an a single phase just below the liquidus temperature, but the phase transformation of a single phase into a α duplex phases occurs below 1320°C. Since the volume fraction of γ phase increases with decreasing temperature below 1320°C, the precipitation of fine γ phase in a matrix phase improves the hot ductility at temperatures between 950 and 1100°C. The sigma phase is known to be formed through the eutectoid decomposition of a ferrite phase into sigma phase at temperatures between 600 and 950°C. It is reported that the precipitation of sigma phase played an important role in improving the ultimate tensile strength and the yield strength while decreasing the ductility as the ageing temperature was increased from 600 to 900º C.

3.4 Effect of ageing on the mechanical properties

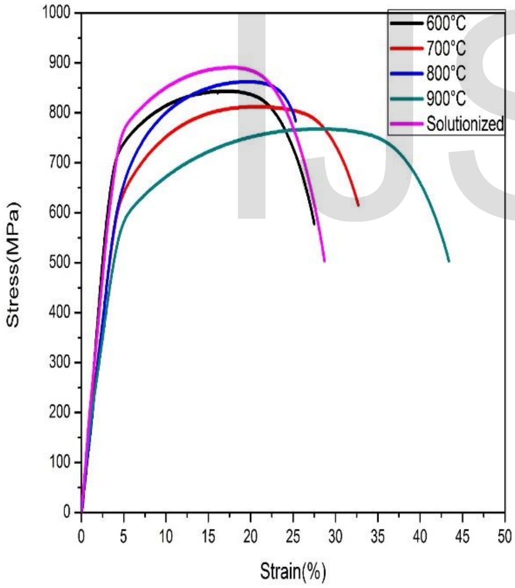

Figure 10 Engineering Stress – Strain plot under different Heat treated condition for 60 minutes

the engineering stress-strain curves for the specimens for all heat treatment conditions. Table 4.2 shows the tensile properties of the material such as tensile strength, yield strength and percentage elongation for all heat treatment conditions. The variation of tensile strength, yield strength and percentage elongation with ageing temperatures is as shown in the figure 4-29 to 4-31. After solutionizing the specimen at 1100^0C temperature for 60 minute, the samples had been subjected to the ageing heat treatment process in order to study the ageing behaviors with respect to mechanical and micro structural characterization. From the figure 4-29 and figure 4-30 we can see Figure shows the engineering stress-strain curves for the specimens for all heat treatment conditions. Table 4.2 shows the tensile properties of the material such as tensile strength, yield strength and percentage elongation for all heat treatment conditions. The variation of tensile strength, yield strength and percentage elongation with ageing temperatures is as shown in the figure 4-29 to 4-31. After solutionizing the specimen at 1100^0C temperature for 60 minute, the samples had been subjected to the ageing heat treatment process in order to study the ageing behaviors with respect to mechanical and micro structural characterization. From the figure 4-29 and figure 4-30 we can see t the both yield strength and the ultimate tensile strength was increasing with increase in the ageing temperature because of the formation of the sigma.

4 CONCLUSIONS

Effect of heat treatment on the microstructural evolution & mechanical properties of Super duplex stainless steel was investigated and based on the results obtained, following conclusions were derived.

1. As the ageing temperature was increased the volume fraction of the sigma phase also increased.

2. Sigma phase formation was mainly due to the eutectoidal decomposition of molybdenum and the chromium atoms in the ferritic phase in temperature range of 600 - 900º C.

3. Sigma phase was usually found at the ferrite/ferrite and austenite/ferrite boundaries.

4. As the ageing temperature was increased, the ultimate tensile strength and the yield strength was increased because the rate of sigma phase precipitation was increasing with increasing temperature.

5. The ductility of the samples was decreasing with increasing temperature because of the embrittlement caused by the sigma phase in the ferritic phase.

6. The strain hardening exponent n, also reached a minimum value of 0.32 and this can be attributed to the formation of the sigma phase precipitation in the ferritic phase which reduced ductility and increased the resistance to the neck formation.

5 SCOPE FOR FUTURE WORK

1. Studying the effect of ageing heat treatment on stress corrosion resistance.
2. The study of effect of initial grain size on the fatigue behavior during aging.
3. A comprehensive study of the influence of alloying elements on thermodynamics and kinetics of aging in the ferritic phase of wrought grades of laboratory melt DSS.

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