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A Study on Precipitation Kinetics of Super Duplex Stainless Steels

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Abstract—In duplex stainless steels ferrite and austenitic phases are present in balanced quantities and are easy to fabricate and having excellent corrosion resistance with higher strength. Duplex stainless steels find major applications in oil and gas industries, chemical process plants, pulp and paper industry and desalination plants. The present study includes the precipitation of intermetallic phases in super duplex stainless steels through frequent aging and heat treatments. The effect of Sigma phase, Chi phase, R phase and 475^0C embrittlement on super duplex stainless steel were studied.

Index Terms—Super Duplex Stainless Steel, Sigma phase, Chi phase, R phase and 475^0C embrittlement.

1 GARDES, SPECIFICATONS AND METALLURGY OF DSS

Major characteristic of duplex stainless steel is high Chromium content (Approx. 20–25.4%) but low content of Nickel (1.4–7%) compare to austenitic grades. For balancing the microstructure Molybdenum (0.3–4%) and Nitrogen additions are essential. Nitrogen is added to increase the strength. Manganese can be added in replacement to Nickel and it also increases the solubility of nitrogen in the material. The below table illustrates the chemical composition of super duplex stainless steel.

TABLE 1
CHEMICAL COMPOSITION OF SUPER DSS

Grade	C	Cr	Ni	Mo	N	Mn	Cu
2507	0.03	26%	6-8%	3-5%	0.32%	1.20%	0.5%

The metallurgical behavior of DSS can be studied in detail with the help of Fe- Cr-Ni ternary phase diagram plotted for 68% iron content [1]. From the above diagram it is clear that Fe- Cr-Ni alloys solidifies as ferrite (α) and some of the alloys transform into austenite (γ) when the temperature reaches 1000^0 C. The ferrite - austenite phase balance in the microstructure can be studied with the help of multi variable linear regressions.

1. Creq = % Cr + 1.73% Si + 0.88% Mo.

2. Nieq = % Ni + 24.55% C + 21.75% N + 0.4% Cu.

3. % Ferrite = -20.93 + 4.01 Creq – 5.6 Nieq + 0.016T.

Where T = Annealing temperature ranging from 1050 – 1150^0 C.

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Nitrogen addition can rapidly increase the rate of transformation of ferrite to austenite by increasing the temperature [2].

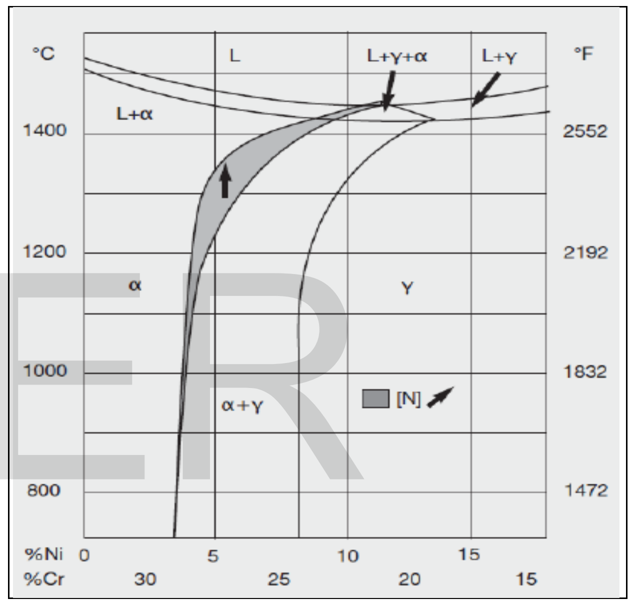

Fig.1. Fe-Cr-Ni ternary phase diagram with 68% iron [1]

3 INTERMETALLIC PHASES IN SUPER DSS

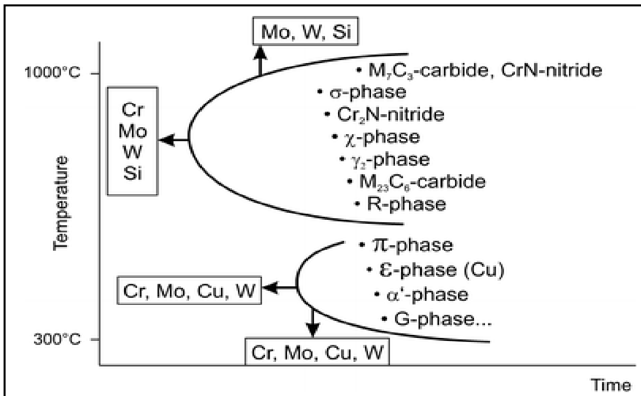

Fig.2. Precipitation of different phases in DSS [3]

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3.1 Sigma Phase

Sigma phase precipitation occurs at temperatures between 600⁰C to 1000⁰C and the phase is rich in Cr and Mo. Sigma phase has strongest influence on corrosion resistance. It increases intergranular corrosion and reduces the pitting corrosion resistance of DSS. Sigma phase has a tetragonal crystal structure with 32 atoms per unit cell and 5 different crystallographic sites [3].

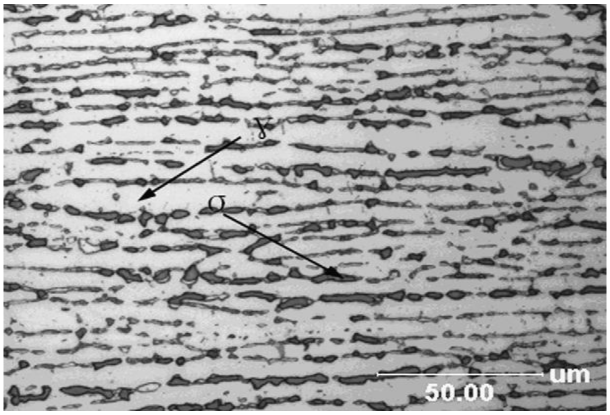

Fig.3. Optical microstructure showing Sigma phase in DSS sample aged at 850⁰C for 48 hours

3.2 Chi Phase

Chi phase is tpologically close packed phase and rich in Fe, Cr and Mo. Chi phase occurs at temperatures around 700⁰C.

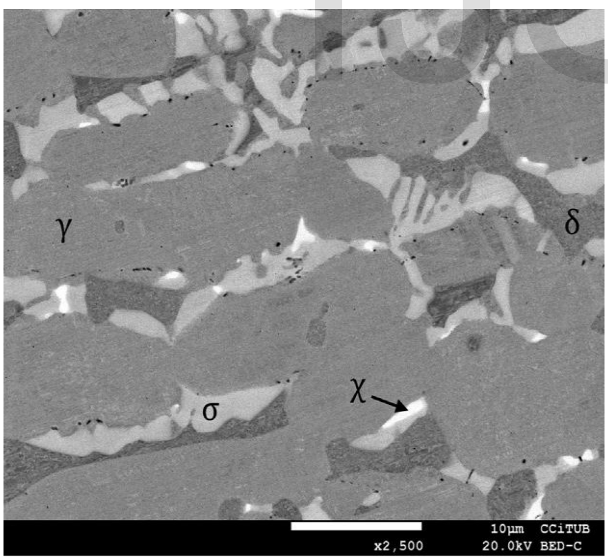

Fig.4. FESEM–BSE image of DSS 2205 aged 1 h at 830°C indicating Chi Phase (Bright Phase)

The Chi phase precipitates at lower temperatures as compared to Sigma phase [7]. It is difficult to study the effect of Chi phase on corrosion and toughness since its often coexists with the Sigma phase [4].

Annealing time	Composition								
	Phase	DSS 2205				SDSS 2507			
		Element (%wt)							
		Cr	Fe	Ni	Mo	Cr	Fe	Ni	Mo
-	Austenite (γ)	21.6	67.4	6.5	2.6	24.9	62.6	8.3	3.3
	Ferrite (δ)	24.9	64.6	4.4	4.6	27.3	60.9	5.9	5.2
10 min	Austenite (γ)	21.4	67.6	7.0	2.1	23.6	62.5	8.5	3.9
	Ferrite (δ)	25.2	65.3	4.1	4.4	26.6	65.8	3.7	3.1
	Sigma (σ)	28.3	57.7	3.8	8.2	30.3	56.7	3.7	8.2
	Chi (χ)	23.3	57.7	3.2	14.2	26.1	54.1	4.3	14.9
300 min	Austenite (γ)	21.1	65.9	6.3	2.4	24.5	62.8	8.9	3.0
	Ferrite (δ)	23.3	67.8	2.7	1.6	21.7	68.9	6.3	1.4
	Sigma (σ)	30.1	56.8	2.5	8.9	30.7	56.2	4.0	7.6
	Chi (χ)	26.9	53.8	2.5	16.3	29.8	53.4	3.7	14.0
540 min	Austenite (γ)	21.7	64.0	7.2	2.7	24.2	60.2	8.0	3.2
	Ferrite (δ)	24.7	69.0	2.1	2.0	20.1	67.9	6.1	2.0
	Sigma (σ)	29.6	59.0	1.8	6.1	31.2	54.2	3.2	8.5
	Chi (χ)	25.1	56.0	2.3	16.5	-	-	-	-

Fig.5. Phase evolution after heat treatment with chemical composition for DSS 2205 and SDSS 2507 [4]

3.3 R Phase

R Phase exists between 550⁰C to 650⁰C temperature range. R Phase is rich in Mo and having a trigonal crystal structure. Formation of R Phase reduces the toughness and critical pitting temperature in DSS.

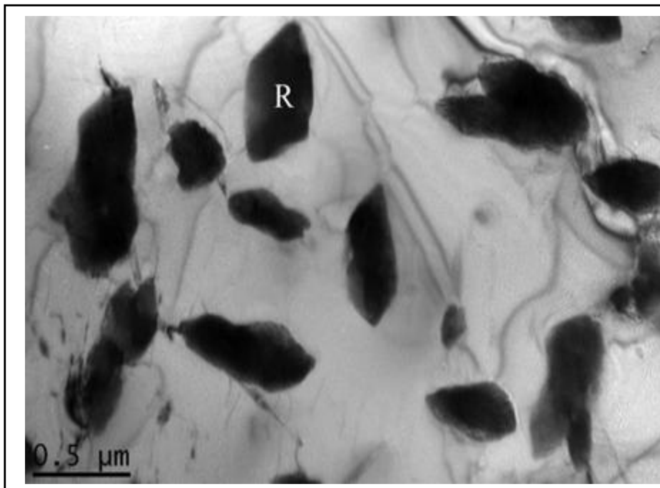

Fig.6. TEM Micrograph showing R Phase in Ferrite Phase

It is observed that as the aging time increases the size of the R Phase also increases [5]. There is a decrement in impact toughness with an increase of volume fraction of R Phase.

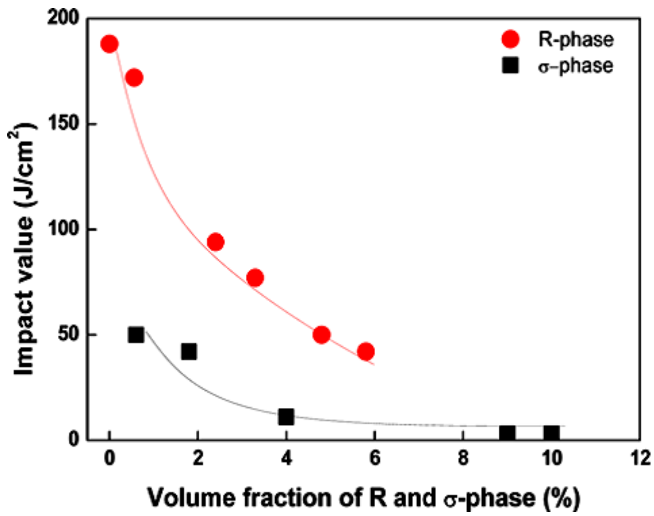

Fig.7. Relation between Impact value and Volume fraction of R Phase and Sigma Phase in SDSS [5].

3.4 475°C Embrittlement

At the temperature ranges from 280°C to 450°C DSS are subjected to thermal aging embrittlement. This will reduce corrosion properties, impact toughness and fatigue properties of DSS [6]. Thermal embrittlement of DSS is caused due to spinodal decomposition in which the Ferrite phase is decomposed into Fe rich α and Cr rich α', this leads to loss in toughness and increase in Ferrite hardness.

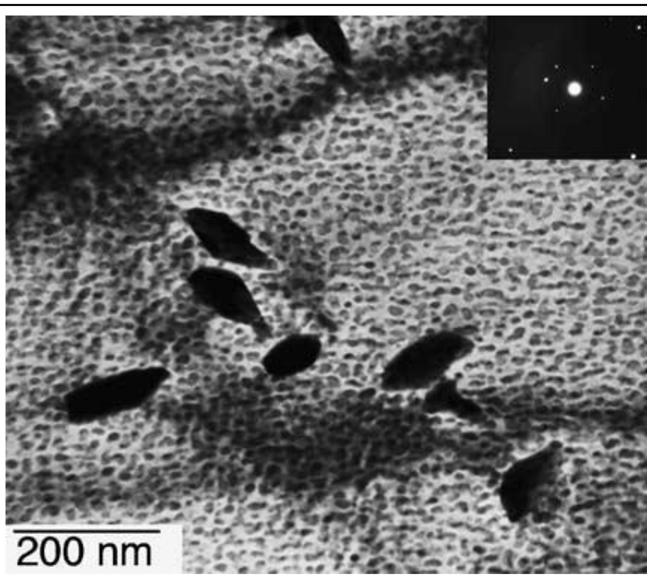

Fig.8. TEM Micrograph showing precipitates present the in Ferritic matrix.

In Fig. 8 precipitates present are Si 2%, Cr 24%, Fe 55%, Ni 7% and Mo 12% respectively.

4 CONCLUSION

From the above study it is evident that the mechanical properties of DSS Can deteriorate by the formation of intermetallic phases. Thus the productions of thick bars or pipes with large diameters are limited because of the formation of Sigma Phase inside the pipe or bar where the cooling rate is relatively less after solution annealing. Chi Phase and Sigma Phases are simultaneously found so it is very difficult to study their individual effect on corrosion resistance and impact properties. The transformation kinetics in super DSS are faster when the percentage of Cr and Mo additions was higher. Impact toughness gradually decreases with increase in R Phase at the time of initial aging and it slowly decreases with an increase in Sigma Phase. The spinodal decomposition was observed to be higher when Ferrite to Austenite ration is equal to 1.5 (α/γ = 1.5)

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