

Mechanical and Microstructural Characterization of Diffusion Bonded Super Martensitic Stainless Steel Joints

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Abstract. Super Martensitic Stainless Steels (SMSS) are low carbon high chromium steels. They possess high strength and resistant to corrosion for fluids like CO₂ and H₂S. Diffusion bonding is a welding technique capable of joining similar or dissimilar metals wherein the solid materials intersperse themselves with time. In this present study, the SMSS specimens were bonded at temperatures ranging from 800-1050°C in the non-inert atmosphere and their microstructure and mechanical properties were analyzed.

Keywords: Diffusion bonding; super martensitic stainless steel; high strength; corrosion resistance; non- inert atmosphere.

1. Introduction

Development of SMSS happened to reduce brittle chromium carbide precipitation on welding of traditional martensitic stainless steels. They are currently produced for the applications of Oil Country Tubular Goods (OCTG). They are based on Fe-Cr-Ni-Mo System with 4-6 % of Ni, 0.5-2.5% of Mo and low carbon contents ($\leq 0.02\%$) [1]. To obtain martensitic lath microstructure SMSS are air-cooled in solution heat treatment temperature at the austenitic field due to their adequate hardenability [2]. Diffusion bonding was an efficient way to manufacture the complex structures with less weight and significant cost savings. The establishment of diffusion bonding process was to manufacture components like honeycomb structures, aircraft engine parts, and hollow engine blades. Diffusion bonding creates permanent joints among materials by establishing interatomic bonds between work pieces through the action of local or general heating or plastic deformation or both [3]. The bonding pressure or pressing load causes the edge to move within the range of atomic force. If there is a molten layer then the decisive factor is the pressing load, which expels the interlayers from the joint. To squeeze out all of the interlayers the pressing load has to be raised. The most stable bonding system was formed by bringing atoms to the distance of minimum energy or equilibrium. Any increase or decrease in inter atomic distance would increase the energy of atomic interaction. Diffusion bonding process was carried out in a bonding chamber in which vacuum is maintained [6]. The vacuum in the chamber reduces the detrimental effect of faying surfaces. The materials to be bonded are to be placed in the chamber with their mating surfaces touching each other and heated to a specific temperature [4-7]. A pressing load was then applied for a specific



amount of time. After the joining was complete, the weldment was cooled in vacuum or quenched depending on the properties of materials involved. The mechanism of diffusion bonding begins with initial point contact of metals, then the contact area increases as the local stress reach the limit of yield stress, then the grain boundary rearrangement happens and the final stage is the volume diffusion of grain boundaries thus the process completes [8].

2. Literature Review

Ayush Barmola.et.al., [8] investigated the effect of recrystallization temperature on super martensitic stainless steel, reported that SMSS on tempering contains tempered martensite and retained austenite with a little amount of α –ferrite present. **Chellappan.et.al.**, [9] discussed the effect of heat input on mechanical and metallurgical properties of SMSS on A-TIG welding found that amount of δ -ferrite increases with the heat input and the tensile strength decreases as the heat input increases and toughness is directly proportional to heat input. **Sanjeev Kumar.et.al.**, [10] discussed the effect of preheat temperature on weldability of martensitic stainless steels reported that the ultimate tensile strength gets increased for the specimen welded at the temperature of 1000⁰ C and preheated at the temperature of 121⁰C. **Surinder Singh.et.al.**, [11] discussed the effects of alloying and heat treatment on properties of SMSS found that While following the heat treatment route, most of the researchers have taken different austenitizing/ heat treatment temperatures (with a wide gap) on which experiments were performed. The temperatures selected either been based on an earlier study or selected randomly without providing the supporting logic. Similarly, some authors conducted heating at different heating rates, with the same limitations as discussed for temperature and very limited work was reported about the magnetic properties of these steels. When reported, it has been stated that austenite is a paramagnetic phase. Thus, the paramagnetic properties were found in the steel until the martensite started to form. However, in some SMSS's that ferromagnetism was observed above M_s temperature (340-230 ⁰ C). The underlying reason behind this unusual behavior was not adequately explained. **Emel Taban.et.al.**, [12] on his discussion about the weldability of super martensitic stainless steels recommends a post weld heat treatment of 620⁰ C-650⁰C for 5 minutes to prevent intergranular stress corrosion cracking. **Kang.et.al.**, [13] on his discussion about the passivity of SMSS found that the passivity combinations of SMSS in both acidic and alkaline environments are Fe oxides and Cr oxides. **Tarun Nanda.et.al.**, [14] on his discussion about the effect of isothermal annealing on microstructure of SMSS reveals that the thermo-mechanical processing route resulted from information of fine austenite grains due to recrystallization of the cold rolled martensitic matrix. The fine austenite grains were much stable at room temperature than the coarse grains. This austenite retention resulted in ductility enhancement. Further, the formation of strain- free grains due to recrystallization resulted in the annihilation of dislocations, which also improved ductility without significantly deteriorating the strength.

A.Dethelfs.et.al., [4] discussed the effect of hybrid diffusion bonding on aluminium tube to sheet connections in the coil wound heat exchangers, found that the weld microstructure, as observed by optical microscopy, consisted of three zones: the TMAZ, the HAZ, and non-affected BM. The TMAZ undergoes significant mechanical deformation under temperature and was characterized by fine equi-

axed grains. The heat affected zone was characterized by a gradual grain growth and an ill-defined boundary around the unaffected base metal. **Zhang Guoge.et.al.**, [14] on his discussion about the solid-state diffusion bonding of Inconel 718 to 17-PH stainless steel reported that a continuous intermetallic film forms along the joint interface when bonding Inconel alloy 718 with 17-4 PH stainless steel at 1000⁰ C for 60 min, with pressure ranging from 16 MPa to 48 MPa. The formation of such phases was caused by the agglomeration of Nb and Ti. In all joints, failure occurred at the interface, nearly without plastic deformation. The maximum tensile strength of the joint reached 774 MPa, more than 70% of that of 17-4 PH stainless steel (1007 MPa). The influence of pressure on joint tensile strength was more significant than that in area reduction. **N.L.Loh.et.al.**, [15] discussed the effect of diffusion bonding of ceramics to metals reported that comparing diffusion bonding to Hot isostatic pressing (HIP) process was considered to be more effective and they are further researching on diffusion bonding using the hot isostatic pressing technique. **M.Balasubramaniam** [16] while discussing the application of box benken design in diffusion bonding of Ti-6Al-4V with 304 stainless steel states that the bonding strength increases with increase in bonding temperature. The increase in bonding strength can be attributed to the formation of intermetallics like Ti-Ag at the interface. **M. Mazar Etabakei.et.al.**, [17] on his discussion about diffusion bonding of Al/Mg₂Si metal matrix composite with AZ91D magnesium alloy at different heating rates state that the average shear strength of the bonded joints showed higher value at lower heating rate. **Kemal Aydin.et.al.**, [18] on his discussion about diffusion bonding of copper to titanium substantiates that suitable weld parameters should be selected carefully. Otherwise, the material with low yield strength would be subjected to a deformation due to the high temperature and pressure. The high temperature lead to grain growth.

From the literature survey it was found that, there was no sufficient literature on diffusion bonding of super martensitic stainless steel, and the novelty of this particular study was the efforts, which had been taken to bond SMSS specimens in non- inert atmosphere in variation to the actual process.

3. Experimental Procedure

The SMSS work pieces to be diffusion bonded are brought as rectangular bars of dimension 195x20x6mm and then cut into pieces of dimension 20x20x6 mm and they were polished by following sequence of steps like surface grinding of the work piece and polishing them using various grades of emery sheets and disc polishing. The hardness of the work piece was measured using Brinell's hardness scale, which showed the value of 66.3 BHN. The chemical composition of SMSS specimen are given in Table 1

Table 1 Chemical composition of SMSS

ELEMENT	Cr	Ni	Fe
COMPOSITION (%)	13	6	81

Tool steel was used as the die material because their suitability comes from the distinctive hardness, resistance to abrasion, deformation and their ability to hold their cutting edges at elevated temperatures. They have the carbon content between 0.5%-1.5% and their four major alloying elements are tungsten, chromium, vanadium, and molybdenum. The modeling and design of the die were done in AutoCAD and CREO parametric. To obtain the desired accuracy of the die was machined using Electric Discharge Machining (EDM). The specimens were placed inside the die and the die was placed inside the muffle furnace and the experiments were carried out at temperatures ranging from 800-1050⁰C.. Figure (1.1) and Figure (1.2) shows the images of the die and the experimental setup.

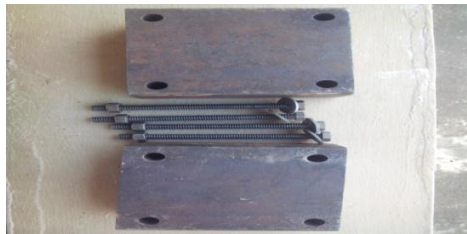


Figure 1.1. Die with the screws



Figure 1.2. Experimental set up

4. Results and Discussions

4.1. Bonding Process

Table 2 shows the experimental table for bonding the SMSS specimens. The main parameters taken were bonding time and bonding temperature, which influences the bonding strength of the specimen .

Table 2. Process table

S.NO	Time(min)	Temperature (⁰ C)	Result	Reason
1	45	800	No bonding	Oxidation
2	45	950	Bonding achieved	-
3	45	1050	Bonding achieved	-

Figure 2(a-c) shows the images of the specimen at the trials, while bonding the specimens. Figure 2 (a) shows the failure in the bonding of the specimen due to oxide formation, since the environment was considered as a major factor in determining the strength of the bonded joints the interface contact was optimized by the treatment of the surface to be bonded by mechanical polishing, etching, and cleaning. At 950 ⁰ C and 1050⁰ C perfect bonding was achieved which can be seen from figure 2(b) and 2(c)

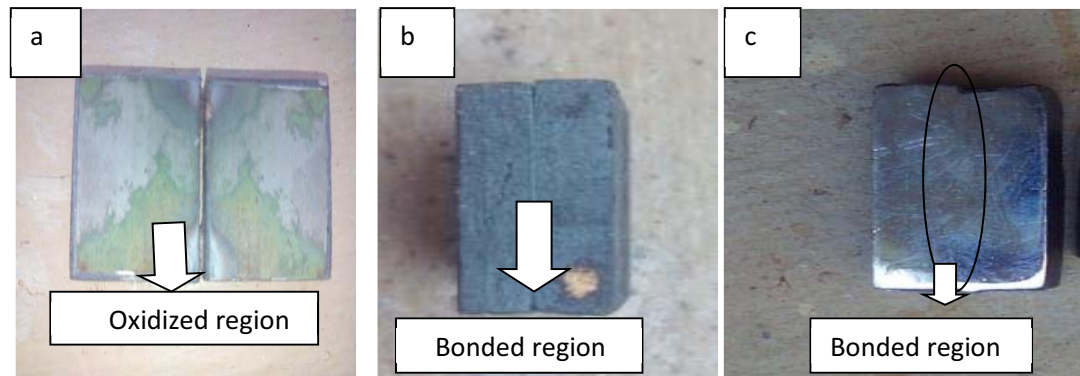


Figure 2. Experimental trials at a) 800⁰ C b) 950⁰C c) 1050⁰ C

4.2 Microstructural Characterization

The microstructure of the base metal, Figure 3(a), shows rolled, pancake structure with varied grain size. The phase mapping structure of SMSS is presented in Figure 3(b). Figure 3(a) shows clearly a well-distinct grain boundary area. The base material has elongated grains of δ -ferrite and streaks (like plates) of δ -ferrite and also randomly dispersed globular particles of Cr_2C_3 [2]. Figure 3(b) shows that the delta ferrite phase in the matrix of martensite is 47% and the alpha ferrite phase, look like to be elongated bands, is 43% [9].

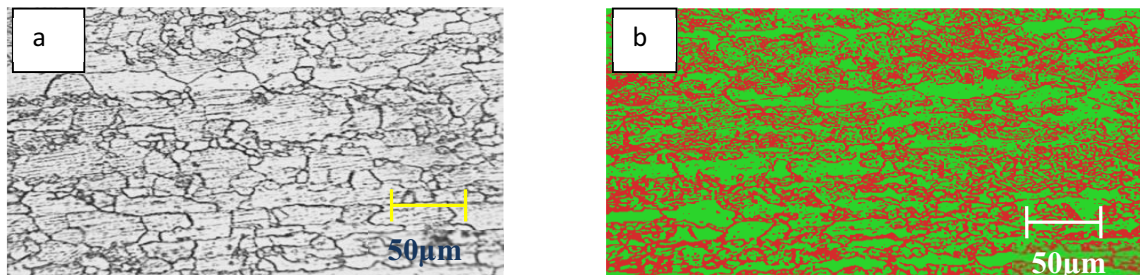


Figure 3. a) Microstructure of base metal

b) Phase mapping image of the base metal

Figure 4(a-c) shows the weld metal microstructure of SMSS specimens at different bonding temperatures. Figure 4(a) shows tree-like structure dark etching martensite in a ferrite matrix along with globular particles are Cr_2 carbides. Figure 4(b) presents, Precipitated particles are Cr_2 carbides which are evenly distributed in a matrix. Figure 4(c) shows martensite in a ferrite matrix, feather type of martensite is observed, but Cr_2C_3 evenly distributed in the matrix [9].

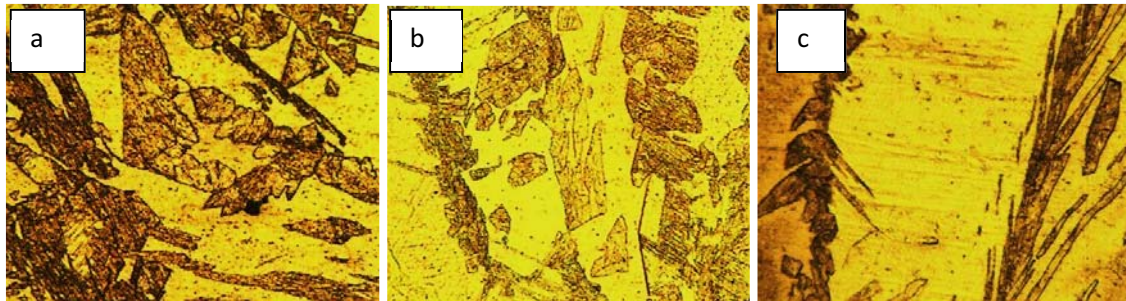


Figure 4. Weld metal microstructure of specimen at a) 800°C b) 950°C c) 1050°C

Figure 5(a&b) shows the microstructure of bonded region. From figure 5(a) it is seen that the perfect bonding has occurred due to diffusion of larger number of atoms to form an uniform structure, as the plastic flow of materials increases. This is highly attributed to increase in heat input because as the temperature increases the grain size becomes smaller and the grain boundaries participating in the diffusion process becomes higher resulting in proper diffusion. From figure 5(b) it is seen that the irregular grain size has resulted in the improper bonding [20].

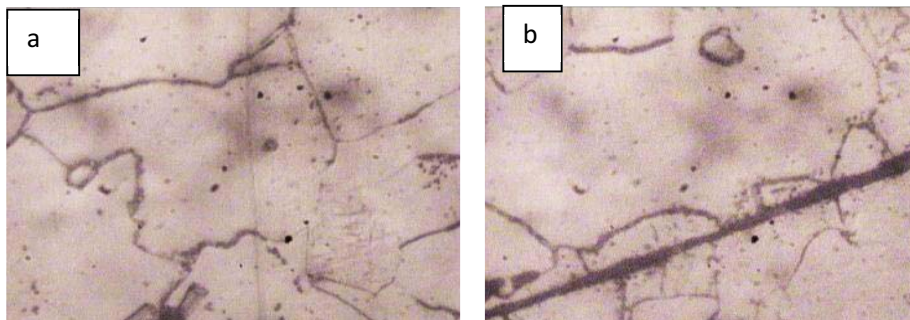


Figure 5. Microstructure of bonded region at a) 1050°C b) 950°C

Table 3 shows the areal fraction of phases present in the weld metal. The analysis was done with the help of phase analysis software and the ferrite content was measured using feritscope. From the table, it is clear that the higher heat input at 1050°C reduces the formation of martensite and the higher cooling rate at 900°C increases the formation of martensite in the weld region [9].

Table 3. Areal fraction of phases present in weld metal

TEMPERATURE(⁰ C)	% OF MARTENSITE	% OF FERRITE
1050	54.61	45.39
950	55.60	43.4
800	67.58	32.42

4.2 Hardness Measurement

The hardness measurements were carried out at different bonding temperatures in weld metal. In each temperature, five measurements were taken and their average values are taken as the result. Table 4 shows the hardness measurements of weld metal. From the table, it is clear that the weld metal hardness value is higher at 800 ⁰ C when compared to the 950 and 1050 ⁰ C. The difference in micro-hardness at 800 and 1050 ⁰ C can be attributed to the differences in the grain size and also major constituents of the martensite phases present at 800⁰ C [2].

Table 4. Hardness measurements on weld metal

TEMPERATURE (⁰ C)	HARDNESS (HV)
1050	224
950	243
800	257

5. Conclusions

Thus the diffusion bonding of super martensitic stainless steels at temperatures ranging from 800⁰ C to 1050⁰ C were done at different time periods and the mechanical and the microstructural characterization were done and the following results were obtained.

- Presence of martensite at lower heat inputs prevents proper bonding of specimens.
- This is contributed to the grain structure of martensite which reduces the diffusion rate and interlocking of grains
- At higher heat input, martensite gets converted into austenite and the grain size becomes finer allowing proper bonding
- Microhardness values decreases at higher heat input (i.e.) at the higher temperature.

6. References

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