Unilag Journal of Medicine, Science and Technology (UJMST) Vol 6. No. 2, 2018

INFLUENCE OF THERMAL TREATMENTS ON SENSITIZATION IN Cr-Mn-Cu AUSTENITIC STAINLESS STEEL WELDS

Amuda*, M.O.H.[1], Lawal, T. F. [2], Enumah, K.S.[3], Ezeonu, L. L.[4] and Onitiri, M.A.[5]

[1-4] Materials Development and Processing Research Group, Department of Metallurgical and Materials Engineering, University of Lagos, 101017
[5] Department of Mechanical Engineering, University of Lagos, Nigeria, 101017

*Corresponding author: mamuda@unilag.edu.ng

ABSTRACT

In an earlier work, Cr-Mn-Cu austenitic stainless steel was established to be susceptible to chromium carbide precipitation in the as-welded condition in the heat input range 207-302 J/mm due to the formation of sensitized structure. Whereas, literature indicate that pre or post-weld thermal treatment has been effective in minimizing carbide precipitation in stainless steel welds; such has not been reported for the welds of the new Cr-Mn-Cu austenitic stainless steel variety. Thus, in this work, sensitization behaviour in Cr-Mn-Cu austenitic stainless steel welds subjected to either pre weld or post-weld thermal treatment was investigated. Pre-weld thermal treatment was conducted with oxy-acetylene flame torch to pre-heat temperatures of 180, 220, 260, 300 and 350^{0}C, respectively for 30 min each and then welded at a heat input of 293 J/mm. Post-weld thermal treatment was conducted on welded coupons at a fixed temperature of 650^{o}C for varying times of 30, 60., 120, 180 and 240 min, respectively. Microstructural characterisation using 10% oxalic acid etch established that post-weld thermal treatment could not prevent the occurrence of sensitization irrespective of the treatment time. On the other hand, pre-weld heat treatment of Cr-Mn-Cu austenitic stainless steel was only effective for eliminating sensitization at preheat temperature around 300^{o}C and above. The work further showed that the mechanism of chromium back diffusion known to be responsible for the effectiveness of post-weld thermal treatment in the control of carbide precipitation in other grades of stainless steel is not applicable to Cr-Mn-Cu austenitic stainless steel grade.

Keywords: Austenitic Stainless Steel, Chromium Back-diffusion, Chromium Carbide Precipitation, Pre-weld Thermal Treatment, Post-weld Thermal Treatment

INTRODUCTION

Few decades ago, the stainless steel community introduced a lower grade of the austenitic variety into the market to replace the standard nickel-chromium grade (International Stainless Steel Forum (ISSF), 2005; Coetzee and Pistorious, 1996). This lower grade, developed by substituting nickel for manganese, was motivated by the high cost and supply issues associated with nickel, a strongly austenite forming and stabilizing element. Such substitution does equally involve the introduction of either nitrogen (N) or copper (Cu) to further enhance the austenite phase stabilization capacity of the manganese (Urade and Ambade, 2016; Charles *et al*., 2010; Charles *et al*., 2009). Thus, there are two distinct compositions of the new grade of austenitic stainless steel-either Cr-Mn-N or Cr-Mn-Cu-; and they are commercially available as either 202 or 204 series, respectively. These new lower grades of the austenitic variety particularly the Cr-Mn-Cu sub-grades are affordable; and consequently, are being deployed as competitive alternatives in industries using the standard Ni-Cr variety. Such industries traverse chemical, electrical machinery/equipment, pharmaceuticals and medicine, transportation, defense and armoury, food and beverages, water supply and household consumables (Boniardi and Casaroli, 2014; Baddoo, 2008).

In many of the identified industries, fusion welding is the standard technology for the construction of the integrated infrastructure deploy; and in maintaining their operations. But, the after-weld characteristics of the materials are often negatively affected by the welding heat. This is because welding process takes the material through a range of transformations involving both the resolidified zone and the heat affected base metal. This induces differential microstructural characteristics in the material (Amuda, 2011; Lippold and Kotecki, 2005). A major consequence of welding heat induced microstructural changes in stainless steel is the precipitation of a second phase particularly chromium carbide. Such precipitation depletes the matrix of the stoichiometry chromium needed for the prevention of corrosion. So, stainless steel with such condition is considered sensitized and as such, cannot offer the needed corrosion resistance (van Warmelo *et al*., 2007).

The phenomenon of sensitization in stainless steel has been widely studied (Yan *et al*., 2010; Lee *et al*., 2009; van Warmelo *et al*., 2007; du Toit *et al*., 2007). du Toit *et al*. (2007) reported that sensitization in stainless steel particularly the ferritic grade occurs via four modes. Two of these modes are related to the cooling rate associated with the heat input. They established that rapid cooling from low heat input may sensitise the weld as well as slow cooling from excessively high heat input. They therefore recommended that, to avoid sensitisation during the welding of ferritic stainless steel, the heat input should be within the range 0.5 - 1.5 kJ/mm. Arising from this recommendation, Amuda and Mridha (2011) investigated sensitisation in thin gauge AISI 430 ferritic stainless steel welds and established that heat input greater than 432 J/min (corresponding to arc current and electrode traverse speed in the range 70 - 90 A and 3 mm/s – 3.5 mm/s, respectively) provides safe welding parameters for avoiding sensitisation in such welds.

Literature is scarce on the existence of such parameters for the welding of thin gauge austenitic stainless steel; more specifically, for the new Cr-Mn-Cu austenitic variety. Therefore, in an exploratory work, Amuda *et al.* (2016) adopted the range of welding parameters established for safe welding of thin gauge AISI 430 ferritic stainless steel to investigate sensitisation behaviour in the new Cr-Mn-Cu austenitic stainless steel welds. Sensitization behaviour was evaluated in terms of the precipitation of chromium carbide in the heat affected zone (HAZ) of the welds. The work established that Cr-Mn-Cu austenitic stainless steel weld was susceptible to chromium carbide precipitation in the as-welded condition in the heat input range 207-302 J/mm due to the formation of sensitized structure. Whereas, literature indicate that pre or post-weld thermal treatment can assist in minimizing the problem of carbide precipitation in stainless steel welds by controlling the mechanism of chromium diffusion as well as moderating the thermal cycle in the welds (Tavares *et al.*, 2014; Kaul *et al.,* 2009; Campbell, 1992). While this has been reported for some ferritic and other grades of austenitic stainless steel welds; such has not been reported for the welds of the new Cr-Mn-Cu austenitic stainless steel variety.

Thus, in this work, the influence of pre or post weld thermal treatment on the sensitisation behaviour of Cr-Mn-Cu austenitic stainless steel weld is investigated. This investigation is conducted as a follow-up to the earlier work in establishing strategy for mitigating sensitisation in the new grade of the austenitic variety.

MATERIALS AND METHODS

The grit blasted 3 mm thick stainless steel material, procured from Owode Onirin International Steel Market, Lagos, Nigeria on GPS 6° 36' 38.88" E 3° 25' 8.039", was cut into coupons of dimension 150 mm x 75 mm. This was followed by deep agitation in a solution of benzene to remove adhering dirt and grease. The chemical composition of the material presented in an earlier work (Amuda *et al.*, 2016) indicated that the material was a Cr-Mn-Cu austenitic stainless steel containing those elements at 14.89, 10.15 and 1.12%, respectively with Ni at 0.34%. This range of composition corresponds to the new series of austenitic stainless steel with extra low nickel where N has been replaced with Cu and Ni substituted for Mn to reduce cost and improve workability (ISSF, 2005).

Afterwards, autogenous full bead on plate weld was produced using direct current straight polarity (DCEN) arc torch from a Tungsten Inert Gas (TIG) machine at a constant voltage of 20V. The specific combination of arc current and electrode traverse speeds adapted from Amuda *et al*. (2016) used for the welding is presented in Table 1. The heat input was estimated using the relationship provided elsewhere (Amuda and Mridha, 2011).

Table 1: Process parameter combination adopted for full bead on plate weld

Weld Coupons	Voltage (V)	Current (A)	Welding Speed (mm/min)	Heat Input (J/mm)
WC 1	20	50	95	302
WC 2	20	90	177	293
WC 3	20	150	417	207

Pre-weld thermal treatment was conducted on 10 coupons before welding using neutral oxy-acetylene flame at temperatures of 180, 220, 260, 300 and 340^{o}C, respectively for 30 min; while another sets of 5-welded coupons were subjected to post-weld thermal treatment in a muffle furnace kept at a temperature of 675^{o}C for various holding times of 30, 60., 120, 180 and 240 min, respectively. The details of the thermal treatment conditions are presented in Table 2.

Table 2: Parameters for thermal treatment of Cr-Mn-Cu austenitic weld coupons

Material	Heat Input (J/mm)		Coupons' Treatment Condition									
			Pre-weld					Post-weld				
WC 2	293	Temp.(^{o}C)	180	220	260	300	340	650				
		Time (Min)	30					30	60	120	180	240

The as-welded coupons and those thermally treated were sectioned transverse to the welding direction and prepared for metallographic procedures using standard technique as described in the literature (Vander Voort, 1999). The microstructure of the un-welded base metal was characterized by immersion etching in a solution of aqua regia (100 ml HCl + 33 ml HNO_3 + 100 ml C_2H_5OH) before sensitization test. Sensitisation in both the as-welded and thermally treated samples was investigated using an *in-house* developed kit conforming to the procedure in ASTM A262 Practice A (2004) standard. The details of the circuit diagram for the *in-house* purpose-built rectifier and the rectifier are presented in Amuda et al. (2016).

RESULTS AND DISCUSSION

Microstructure of Base Metal

Amuda *et al*. (2016) reported that the microstructure of the base metal Cr-Mn-Cu austenitic stainless steel re-presented in Figure 1 consisted of an equiaxed fully austenitic matrix with some dispersed carbides inter and intra-granularly within the austenite grains. This microstructure is typical of annealed austenitic stainless steel material. Similar microstructural morphology was previously reported in the base metal of unwelded AISI 202 austenitic stainless steel by Sudhakaran *et al*. (2014); suggesting that the Cr-Mn-Cu austenitic stainless steel belongs to the 200 series grade.

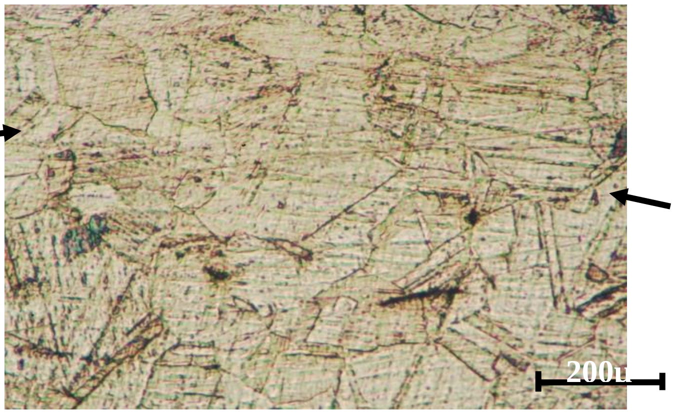

Figure 1: Optical micrograph of un-welded Cr-Mn-Cu austenitic stainless steel base metal (Amuda *et al*., 2016)

Topography and Microstructure of Thermally Treated Resolidified Welds

The topographies of both pre-weld and post-weld coupons presented in Figure 2 show a smooth continuous weld track without any physical defects such as melt-through or cracks similar to the topography of the non-thermally treated welded coupons in Amuda *et al*. (2016). However, the surface appearances in the differently thermally treated weld coupons show clear contrast. The pre-weld coupons (Figure 2a) exhibit bright surface arising from the neutral flame of the oxy-acetylene; whereas, the post-weld coupons exhibit grey-dark surface (Figure 2b) owing to the formation of oxide scale on the coupons which is attributed to the oxidising environment of the furnace atmosphere during post weld thermal treatment.

(a)

(b)

Figure 2: Topographies and surface appearances of: (a) pre-weld and (b) post-weld coupons

The microstructure of the resolidified weld coupons shown in Figure 3 consists of austenite matrix with sparse distribution of vermicular (feathery) δ-ferrite. du Toit (1999) reported similar primary solidification structure in chromanite^{TM} austenitic stainless steel welds. The microstructure in the as-welded control coupons (Figure 3a) consists of large austenite grains with discontinuous distribution of ferritic phase as discrete islands within the dendritic austenite structure. These microstructural features are equally prevailing in both the pre-weld and post weld coupons but with differential grain morphology.

The grain structure in the fusion zone (FZ) in the as-welded coupon is elongated in the direction of solidification while those in the high temperature heat affected zone (HAZ) are essentially equiaxed (Figure 3a). The grain morphology at the weld interface consists of a mixture of elongated grains growing into the FZ and equiaxed grains developing towards the high temperature heat affected zone (HTHAZ). The dynamics of the grain morphology at the weld interface is influenced by the progression of solidification and cooling rate. Therefore, the interface is a moving one with no fixed morphology at any instant until solidification and cooling is complete. But in the pre-weld coupons, the grain is coarse particularly in the HTHAZ (Figure 3b). This is in consonance with the trend expected of pre-thermally treated welds as pre-heating reduces the cooling rate and extends the time spent above the grain coarsening temperature which results in grain growth.

The grain structure in the post-weld thermally treated coupons, however, exhibit a refined grain morphology unlike what was obtained in the pre-weld samples. The post weld thermal treatment at 650^oC appears to facilitate the precipitation of second phase at the grain boundaries which has a pinning effect on the grain structure in the HTHAZ. This restricts grain coarsening resulting in the refined grain structure in Figure 3c.

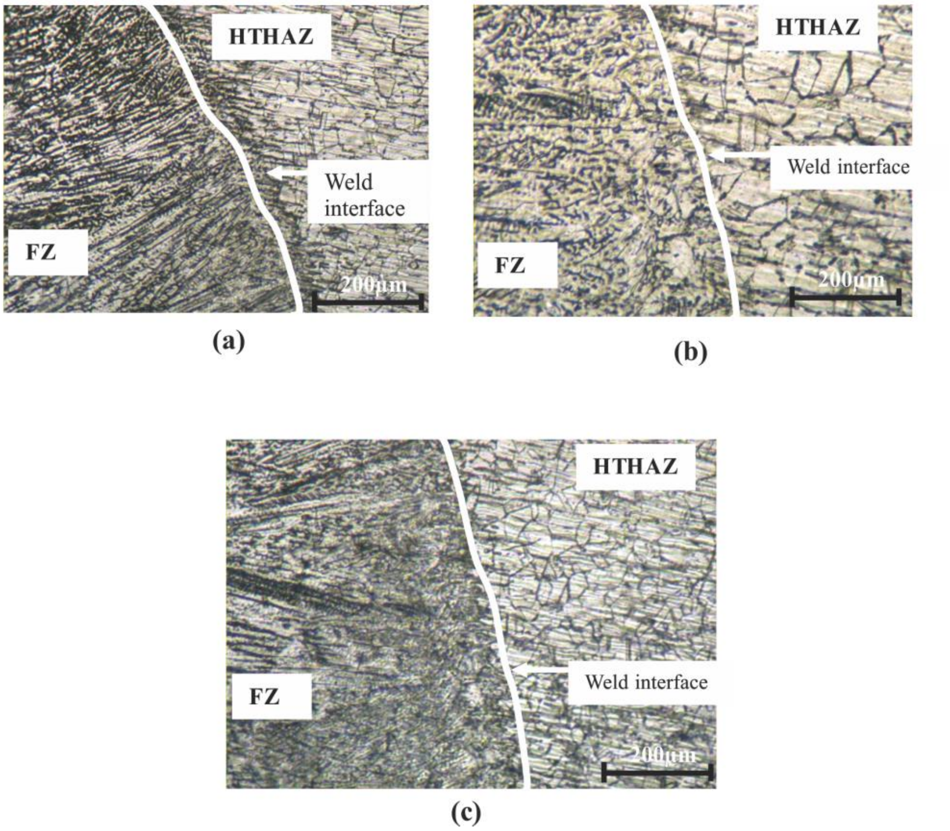

Figure 3: Optical micrograph of resolidified weld coupons differential grain morphology in the HTHAZ: (a) control weld, (b) pre-weld thermally treated at 260^oC for 30 min and (c) post-weld thermally treated at 650^oC for 30 min

Sensitization Behaviour in Thermally Treated Weld Samples

ASTM A262 Practice A (ASTM 262, 2004) provides screening criteria for identifying sensitized structure after oxalic acid etch test depending on the state of the grain boundary. These criteria described in Amuda *et al*. (2016) are invoked in analysing the influence of pre-weld or post weld thermal treatment on the sensitization behaviour of Cr-Mn-Cu austenitic steel welds.

Preliminary sensitization analysis of the weld coupons produced with the parameters listed in Table 1 showed that the coupon produced with heat input of 293 J/mm exhibited strongly ditched structure relative to those produced at heat input of 207 and 302 J/mm. Therefore, it was selected as the control sample for the study of the influence of thermal treatment on the sensitization behaviour of Cr-Mn-Cu austenitic stainless steel welds. The microstructure of the control weld obtained after 10% oxalic acid etch shown in Figure 4a indicates clearly ditched structure in which all the grains are completely surrounded with ditches. Such strongly ditched structure is suggestive of susceptibility to chromium carbide precipitation which is a measure of sensitization.

Figure 4b-f are the microstructural features of weld coupons, preheated at temperatures of 180, 220, 260, 300 and 350^{0}C before welding, after 10% oxalic acid etch. The figure shows that weld coupons pre thermally treated at temperatures below 300^{o}C exhibit strongly ditched structure with apparent coarsening of grain structure at higher preheating temperatures (Figure 4b-d). But, those pre-thermally treated at 300 and 340^{o}C exhibit no such ditch structures. Furthermore, no single grain in the microstructures is either partly or completely surrounded by ditches. These microstructural conditions at the pre-treatment temperatures in relation to the criteria established for the identification of sensitized structure in stainless steel welds suggest that those pre-thermally treated at temperatures below 300^{o}C are sensitized while those pre-thermally treated above 300^{o}C are sensitization free.

Literature established that pre heat treatment assist in reducing the thermal gradient in the weld resulting in lower cooling rate particularly in the HTHAZ (Alkali *et al*., 2014). This inhibits martensitic transformation in the austenitic material and facilitates transformation to quasi-equilibrium condition involving precipitation of second phase such as chromium carbide (Lee *et al*., 2009). But in the present investigation, it is evident that pre-weld thermal

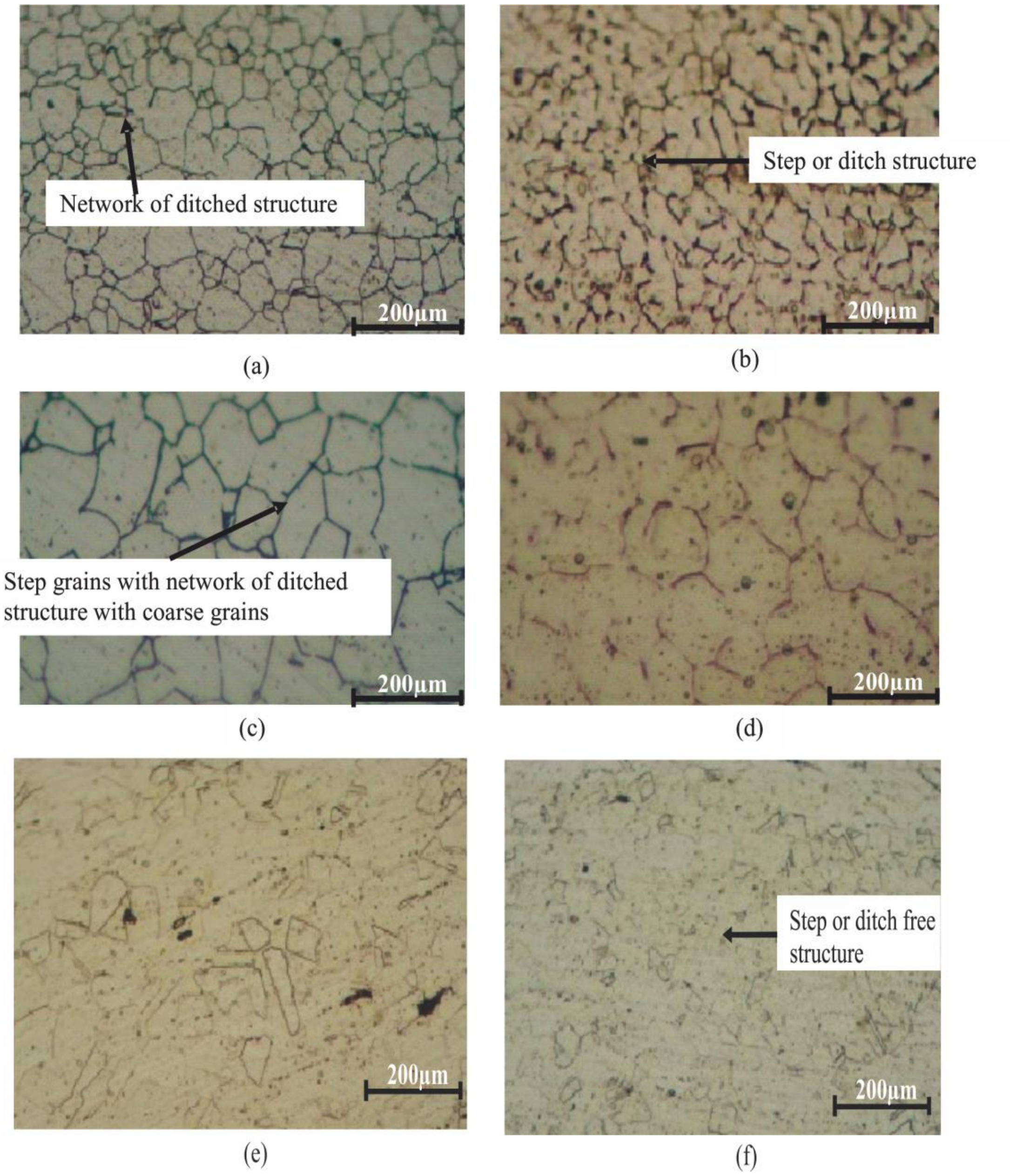

Figure 4: Microstructural features within the HTHAZ of welds preheated at different temperatures after 10% oxalic acid etch: (a) control weld, (b) 180^{o}C, (c) 220^{o}C, (d) 260^{o}C, (e) 300^{o}C and (f) 340^{o}C

treatment is only effective at specific temperature threshold in preventing the onset of sensitization in Cr-Mn-Cu austenitic stainless steel welds. This is because at temperatures below 300^{o}C, the solubility of carbon in austenite is quite insignificant but pick-up at temperatures around 300^{o}C (Boniardi and Casaroli, 2014). Thus, austenite at pre-heat temperature below 300^{o}C is unable to lock-in carbon in solution, and this release carbon freely to combine with chromium in the matrix forming chromium carbide. But, at 300^{o}C and above, the solubility of carbon in austenite picks up and this locks carbon in solution depriving the matrix of free carbon. The absence of free carbon to combine with chromium is essentially responsible for the sensitization-free structure obtained in Figure 4e and 4f.

The microstructural features of weld coupons post-weld thermally treated at 650^{o}C for varying times of 30, 60, 120, 180 and 240 min, after 10% oxalic acid etch, shown in Figure 5b-f in relation to the control weld (Figure 5a) show extensively ditched structures across all the treatment conditions. The grains are surrounded by network of ditches indicating a strongly sensitized structure. The figure further demonstrates that grain growth occurred in the welds with increasing post-weld soaking times. While literature indicate that post-weld thermal treatment at around 650^{o}C is effective in improving sensitization behaviour in Ni-Cr austenitic stainless steel welds, the findings from the present investigation is suggestive of a contrary effect in Cr-Mn-Cu austenitic variety. For instance, Punburi and Tareelap (2013) reported that post weld thermal treatment of 304 austenitic stainless steel and 430 ferritic stainless steel welds at about 900^{o}C for 36 hours followed by water quenching prevented the precipitation of chromium carbide and equally replenished chromium depleted zones via chromium back diffusion. However, in the work of Tavares *et al*. (2014), it was established that post weld treatment of supermartensitic stainless steel welds at 650^{o}C for 30 min encouraged the precipitation of metallic carbides. This contrasting finding, other than the difference in the treatment temperature, may be attributed to the subsequent quenching undertaken by Punburi and Tareelap (2013) which was not undertaken by Tavares *et al*. (2014). Though, post weld thermal treatment improves diffusion of chromium to facilitate chromium back diffusion, this also enhances the migration of carbon to the grain boundary resulting in the precipitation of chromium carbide**.**

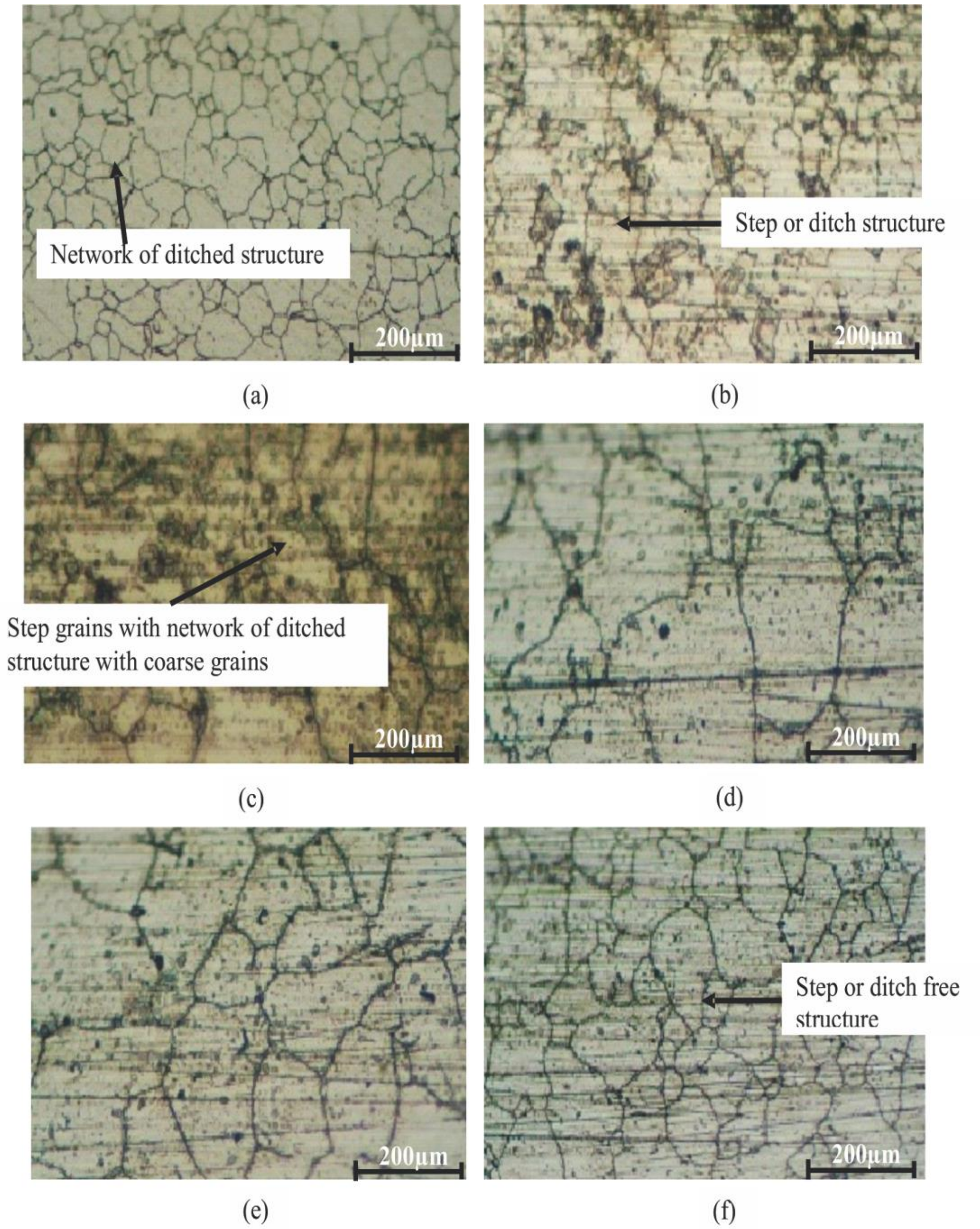
Network of ditched structure
200µm
(a)
Step or ditch structure
200µm
(b)
Step grains with network of ditched structure with coarse grains
200µm
(c)
200µm
(d)
200µm
(e)
Step or ditch free structure
200µm
(f)

Figure 5: Microstructural features within the HTHAZ of welds post-weld thermally treated at 650^oC for varying times: (a) control weld, (b) 30 min, (c) 60 min, (d) 120 min, (e) 180 min and (f) 240 min

Therefore, the subsequent quenching of the post-weld thermally treated welds by Punburi and Tareelap (2013) locked carbon in solution in the austenite phase. This ensures that while chromium depleted zones are replenished via chromium back diffusion, carbon is not freely available to initiate the precipitation of chromium carbide. The findings from the present investigation aligns with that of Tavares *et al.* (2014) that post-weld thermal treatment of welds encourages continued depletion of chromium from the matrix thus facilitating the development of sensitized. It is suggestive that this may be due to the absence of a subsequent quenching process after post-weld thermal treatment. The absence of such process permits uninhibited interaction between chromium and carbon resulting in the development of sensitized structure observed in Figure 5b-f. This has shown that the generally accepted chromium back diffusion mechanism for replenishing chromium depleted zone is not applicable in the present investigation.

CONCLUSION

The influence of either pre-weld and post weld thermal treatment on sensitization behaviour in thin gauge Cr-Mn-Cu austenitic stainless steel welds has been investigated. Microstructural characterisation using 10% oxalic acid electrolytic etch established that pre-weld thermal treatment is only effective for preventing sensitization at treatment temperature above 300^oC. At this temperature, carbon is locked up in solution in the austenite phase due to improved solubility which deprived the matrix of free carbon. On the other hand, post-weld thermal treatment is not effective for avoiding sensitization irrespective of the heat treatment time. Furthermore, the work showed that the mechanism of chromium back diffusion which has been responsible for the effectiveness of post-weld thermal treatment in the control of carbide precipitation in other grades of stainless steel is not applicable to Cr-Mn-Cu austenitic stainless steel grade.

ACKNOWLEDGMENT

The technical staff of the Central Workshop, Faculty of Engineering, University of Lagos, Nigeria are appreciated for providing technical assistance for the welding of the coupons. Engineer Hisham Muhammed of Electrical and Electronic Engineering, University of Lagos is equally acknowledged for his input in the development of the circuit diagram.

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Responses to Reviewer Comments on Paper Ref No_17_ENG_121

S/N	Reviewer's Comments	Responses
1.	i. What informed your choice of pre-weld temperatures of 180 – 350°C? ii. Do you think the temperature range is sufficient to cause any significant change in microstructure of the weld zone?	i. The pre heat temperature range is the window generally considered for stainless steel materials for controlling cooling rate. ii. Pre-heating in this instance is not to change microstructure but control cooling rate which influences diffusion and hence sensitisation.
2.	i. At what temperature was the weld done? ii. For Cr-Mn-Cu austenitic stainless steel, what is the standard weld temperature?	i. In fusion welding, this is minimally taken to be the melting point of the materials. It is standard practise in the body of knowledge not to state it. Because there cannot be fusion joining if the materials do not get to melting point; so it is usually implied. ii. The same justification applies in this instance.
3.	Include a table of the chemical composition while stating the source.	Once the source is provided as part of the procedure; it is the rule of thumb not to provide it anymore wholesomely particularly if the source is from the same author(s). Otherwise, the manuscript would be liable to a valid charge of self-plagiarism. What can be done at best is to provide a summary of what was in the source and that has been done in the manuscript.
4.	Did you carry out sensitization test on the welded sample alone? Or do you have samples that were subjected to sensitization without thermal treatment? If you have, where are the results and if not why?	Sensitization is a problem caused by effect of welding heat. Therefore, a weld must be produced before sensitisation test and that was done. The findings were presented in an earlier publication by the authors. This is a follow-up to the previous publication to investigate how sensitisation is affected by either pre or post-weld thermal treatment not by both pre and post-weld thermal treatment. It is either pre or post at a time not both treatments at the same time.
5.	There are editorial works to be done as stated in the manuscript.	Editorial corrections effected as necessary.