EFFECT OF COLD WORK ON HAZ WELD DECAY OF TYPE AISI 304 STAINLESS STEEL

Mohamed Milad, N. Zreiba and J. Ben Nasr*

Tripoli University, Faculty of Engineering, Libya
* National School for Engineers of Sfax, 3038 Sfax, Tunisia
E-mail: Mohamed_m_m2000@yahoo.com

الملخص

تمّ في هذا البحث دراسة تأثير كلا من الشغل على البارد وطاقة اللحام على حساسية (Sensitization) الصلب الاوستينيتي المقاوم للصدأ نوع (AISI304) بحيث تم درفلة عينات من هذا الصلب على البارد لإنقاص سمكها لقيم تصل إلى 50% من السمك الأصلي قبل أن يتم لحامها بطريقة اللحام بقوس التنجستن (TIG). استعملت طريقتان لمعرفة مدى تعرض هذه العينات للصدأ، الطريقة الأولى تعتمد على إظهار البنية المجهرية باستعمال حمض الاكساليك وفقا للمواصفة الأمريكية (ASTM A262), أما الطريقة الثانية فترتبط بمنحنيات الاستقطاب الكهروكيميائي ويرمز لها بالرمز (DL- EPR).

بينت النتائج المتحصل عليها أن تأثير الدرفلة على البارد على حساسية الصلب يكون ملحوظا ومميزا وذا قيمة معتبرة عند نسب الدرفلة من 10% إلى 35% تقريبا. أظهرت النتائج أيضا أنه عند هذا المدى من الدرفلة فإن نسبة الدرفلة الحرجة والتي بعدها يبدأ النقص في مقدار حساسية الصلب للصدأ تتناسب تناسبا عكسيا مع كمية طاقة اللحام. أما عند نسب الدرفلة من 35% إلى 50% تقريبا فإنه لوحظ تأثير ضعيف لكل من نسبة الدرفلة وطاقة اللحام.

ABSTRACT

The weld decay (sensitization) of type 304 stainless steel (SS) is evaluated as a function of prior cold work and welding heat input. Type AISI 304SS is cold rolled to various percentages of thickness reduction up to 50% cold reduction (CR) at ambient temperature before being TIG welded. The susceptibility of 304SS to weld decay is evaluated using ASTM A262 practice A and electrochemical potentiokinetic reactivation (EPR) tests.

The results indicate that the influence of pre-welding cold work on the degree of sensitization (DOS) appears to be distinct and appreciable within the range of 10% to 35%CR. In addition within this range the critical level of cold work after which the DOS starts decreasing is found to be inversely related to the amount of welding heat input. Meanwhile, at higher levels of deformation approximately $\geq$ 35CR%, the results indicate that neither the cold work nor the welding heat input significantly influences the amount of DOS. Analysis of the reactivation scans obtained after the EPR test demonstrate that the DOS is almost independent of the activation current density (Ia), rather the reactivation current density (Ir) mostly influences the results. Microstructural investigation shows that the priority of the intergranular corrosion (IGC) appears to be at austenite grain boundaries and less likely at ferrite / austenite interfaces of the weld metal. Meanwhile, transgranular attack at austenite bulk matrix along the defects is rarely observed.

KEYWORDS: Cold work; Type AISI304 stainless steel; Weld decay; electrochemical potentiokinetic reactivation.

INTRODUCTION

In constructing many apparatus, forming and welding are indispensable processes since the sections to be welded are commonly worked locally when deformed into the final desired shape. As a result, the material experiences a thermomechanical effect in which the interaction between temperature and strain exists so that its functional properties are altered.

Type AISI 304SS is a common material of construction, as it offers good combination of mechanical strength, fabricability and general corrosion resistance. Although 304SS is readily weldable, two major problems namely hot cracking of the weld metal and sensitization of the heat affected zone (HAZ) are often encountered. Sensitization of stainless steel owing to welding is known as weld decay. It is a type of intercrystalline corrosion normally proceeds along both sides of a zone that is parallel to the weld bead and a few millimeters away from it.

Several methods have been adapted to control and minimize weld decay of stainless steel weldments namely:
Employing a solution annealing heat treatment, lowering carbon content or adding stabilizers. However, these methods are either costly or some times difficult to apply. A practical and inexpensive method for reducing sensitization of SS weldments is therefore very much desirable.

Cold working of SS results in microstructural changes such as dislocations, stacking faults, vacancies… etc. The presence of such defects may reduce the susceptibility of the alloy to sensitization since they may provide favorable precipitation nucleation sites for carbides thus eliminating the risk of intergranular corrosion from being proceeded to grain boundaries.

The influence of different kinds of cold work (CW) like tensile straining, forging, rolling, drawing … etc has been referenced in many reports, see for example [1-5]. From such reports one can realize that cold work has a significant influence on the sensitization behavior of stainless steel. However, there is some controversy among the previous investigations, where as some workers indicate a beneficial role of low levels of CW on the sensitization characteristics of SS, others report opposite. Nevertheless, threshold value of CW below which sensitization of SS would be minimized is still questionable and a universally accepted conclusion relating the influence of CW on intergrenular attack of SS is yet to be made. Consequently, the present work addresses the influence of prior plastic deformation in the degree of sensitization of 304SS welded specimens using three welding heat inputs namely 10, 15, and 23 kJ/cm.

EXPERIMENTAL PROCEDURE

Material

The material used in this investigation is a commercial grade type 304 austenitic stainless steel having the chemical composition given in Table (1).

Table 1: Chemical Composition of 304SS Used in This Study

Element	C	Mn	Cr	Si	Ni	S	P	Mo	N	Fe
Wt%	0.058	2.068	18.718	0.501	8.982	0.023	0.026	0.392	0.028	Bal.

304SS is submitted in the form of rectangular bars of 1000 mm long, 60 mm wide and 8 mm thick. The steel plates are sectioned into small strips of equal size (120× 60 ×

8 mm). These will be referred to as "as received" specimens and denoted by AS.R. The strips are then solution annealed at a temperature of 1100^oC for a soaking time of 90 minutes before being quenched in 5% iced brine. That is done aiming to eliminate the intergranular attack of the as received 304SS and to have all materials initially at the same condition. The solution annealed specimens will be denoted as SA.

Cold Rolling

The rolling process is carried out at ambient temperature under oil lubrication. The rolling load and the gap between the operating rolls are adjusted to give very small thickness reduction after a series of passes the steel strips in one direction. Ten levels of percentages of thickness reduction (%CR) are achieved (i.e., 5%, 10 %… 50 %).

Welding

A single pass bead–on–plate weld is carried out down to the center of the strip using automatic gas tungsten arc welding machine (Type LUD 320 Sweden made). The specimen to be welded is placed on a 3 mm thick strip of asbestos (non-conductive material) so that the amount of heat dissipated into surroundings could be minimized. The welding parameters were varied so as to generate weld pads with approximately 10,15and 23 kJ/cm heat inputs. The heat input was controlled by varying the welding arc current (I), while keeping the arc voltage (V) and welding speed (s) almost constants.

ASTM A262 Practice A

This test is carried out by electrolytically etching of 1cm^2 specimen surface area at a current density of 1A/cm^2 in 10% oxalic acid solution. The quality of the material is classified according to the following three basic categories: step, dual, and ditch structure as recommended in the ASTM standards [6].

EPR-TEST

Double loop (DL) version is adopted in this investigation for evaluating the IG susceptibility of 304SS weldments. DL-EPR is carried out following the test conditions proposed by A. kashi et al [7]. The specimens are polarized anodically to a potential of +200 mV versus SCE in the passive range. As soon as this potential is reached, the potential scanning direction is reversed to the original corrosion potential (E_{corr}) at the same scanning rate, Figure (1).

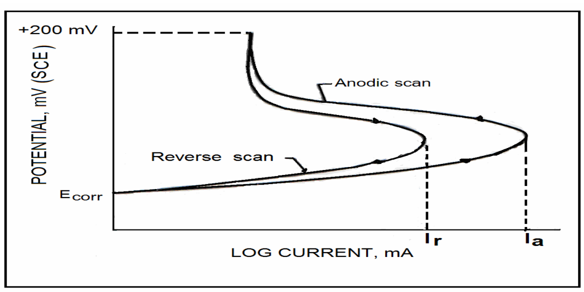

Figure 1: Double loop EPR technique [7]

The maximum current for each loop is measured, Ia for the activation loop, and Ir for the reactivation loop. The ratio Ir/Ia is taken as a measure of the degree of sensitization of the tested sample. The EPR- test conditions are given in Table (2).

Table 2: EPR Test Conditions.

Electrolyte	0.5M H_2SO_4+0.01M KSCN
Temperature	Room Temperature
Reactivation Rate	6V/h
Surface Finish	No. 1200 SiC
Surface Area	1 cm^2

RESULTS AND DISCUSSION

The results of 10 % oxalic acid test show that the microstructure of as received 304SS consists of continuous network of ditches along austenite grain boundaries, Figure (2a). This implies possible chromium depletion and carbide precipitation. From sensitization point of view, this structure would be classified as ditch structure. This is probably because as received material was produced by hot rolling followed by solution annealing heat treatment at 1050C° followed by air cooling. This treatment is not recommended since cooling by air permits sufficient time for the annealed steel to be at the critical sensitization temperature range (450-850C°). Chromium carbide can readily precipitate at grain boundary. Instead the material must have been water quenched so that less chance for carbide to form. The microstructure of solution annealed 304SS using 10% oxalic acid is shown in Figure (2b). This structure may be classified as dual structure.

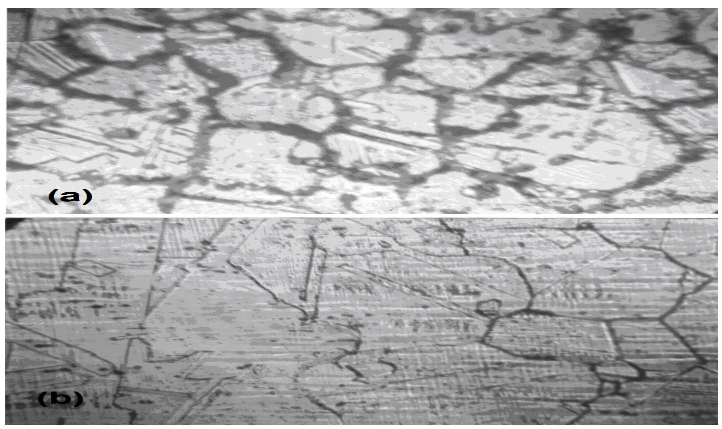

Figure 2: Oxalic acid etches test structure of (a) as received; (b) solution annealed 304 SS, (Magnification 400x)

In this aspect, although complete removal of IG attack is not possible, however, the microstructure of SA 304SS reflects the effectiveness of the solution annealing treatment employed in this study. It is evident that the annealing treatment has significantly depressed the amount of ditches. Both the extent and the width of the attack are substantially reduced, compare micrographs (a) and (b) in Figure (2).

Cold rolling of 304SS produces a new phase which is found to be ά-martensite. ά-phase is a unique feature of austenitic stainless steels induces as a result of cold work or refrigeration heat treatment. Representative microstructures of cod rolled 304SS are shown in Figure (3a, b, and c). It can be seen that the amount of ά- strain induced martensite increases with deformation. On the other hand, no evidence of grain boundary ditches in either microstructures of Figure (3). Accordingly the microstructures of the cold rolled specimens may be classified as step structure.

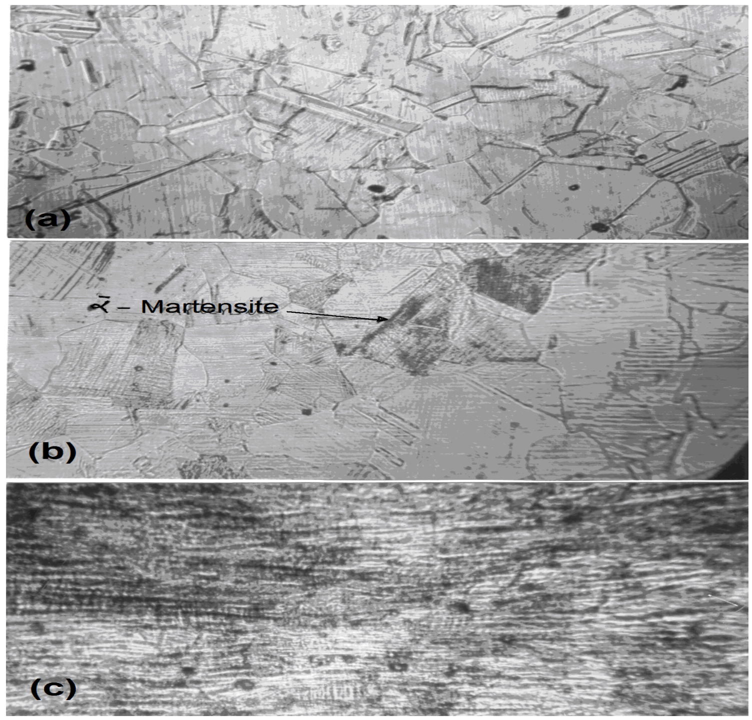

Figure 3: Oxalic acid etch test structure after cold rolling to (a) 10%; (b) 30%; and (c) 50% thickness reduction, (Magnification 200x)

Using 10% oxalic acid test to asses the DOS of 304SS weldments results in the following features:

- Step structure characterized specimens cold rolled 5≤ %CR ≤25 and welded with 10 kJ/cm.
- Specimens of 15%CR and 20%CR experienced ditch structure when welded with 23 kJ/cm
- The rest of the specimen's exhibit dual structure.

From the above observation, it can be concluded that using 10% oxalic acid test does not give good discriminating power in differentiating between the intergranular corrosion of specimens of different conditions. The majority of the specimens show dual structure irrespective of being welded or unwelded. In fact 10% oxalic test is only a qualitative test and is used as a screening test and hence it has to be further supplemented independently by other conventional ASTM standard tests.

Based on the above remarks, the IG susceptibility of 304SS need to be evaluated using DL-EPR technique.

The degree of sensitization of unwelded specimens (as received, solution annealed, and cold rolled) was firstly measured Table (3). As a result, the amount of sensitization produced by welding can be evaluated relative to that presents before welding and comparison can be made on the bases of pre-existing IG attack. Bearing on mind that some ditches still exist in the solution annealed 304SS as estimated by the oxalic acid test.

Table 3: EPR Current Ratio Results for Unwelded and Welded 304SS Specimens

Cold Reduction %	Unwelded Specimens	Welded Specimens		
	Welding Heat Inputs			
%C R	0 kJ/cm	10 kJ/cm	15 kJ/cm	23 kJ/cm
Average Ir/Ia $\times 10^{-3}$				
0(SA)	7.826	8.068	10.137	11.670
5	-	5.027	7.770	10.042
10	1.785	3.115	5.262	6.822
15	-	4.248	10.421	15.983
20	1.972	2.875	12.200	15.695
25	-	4.632	9.886	14.798
30	1.577	6.447	6.823	12.741
35	-	10.231	8.231	8.529
40	1.625	7.962	6.974	9.258
45	-	5.567	8.257	7.096
50	1.040	5.727	7.361	8.727

The variation of DOS as measured in terms of Ir/Ia ratio with % CR of unwelded samples (0 kJ/cm) is shown in Figure (4curve a). Two distinct regions can be recognized in curve a. Of importance, the first region in which the DOS of SA specimen sharply decreases after being initially deformed to 10%CR. Beyond 10 %CR (second region), the DOS becomes almost negligible and independent of cold work. The samples cold rolled ≥ 10% may be theoretically assumed to be free of sensitization that is supposed to be obtained after the solution annealing treatment. Nevertheless, Figure (4) emphasizes the role of solution annealing in reducing the IG attack of AS.R 304SS. actually, this observation confirms the results obtained by the oxalic acid test.

The decrease in the EPR-DOS of SA specimen at CR values above 10% may be attributed to the formation of a distorted structure produced by CW. In this case carbide separation may occur and being randomly distributed among the defects of the deformed structure at deep locations. Consequently, the depleted area (as EPR test concerns with) seams to be masked and being discontinuous. This results in low reactivation current density (Ir) and hence low DOS.

At this stage, two features have to be considered:

- The sequence of CW with respect to heat treatment. Here cold work is performed after the solution annealing which is used to suppress the corrosion attack of AS.R steel. In this case SA 304SS has to be considered as a non-sensitized material. However, if cold work is introduced after a certain sensitization heat treatment or welding, the behavior of 304SS against sensitization will be different.

- The reduction of DOS of SA steel due to 10% CR does not mean that the attack is completely disappeared; rather it has been redistributed and masked amongst the structure defects. However, if the effect of CW is somehow removed, these dispersed carbides may serve as preferred sites for nucleation and growth of new carbides produced by a subsequent sensitization heat treatment or welding.

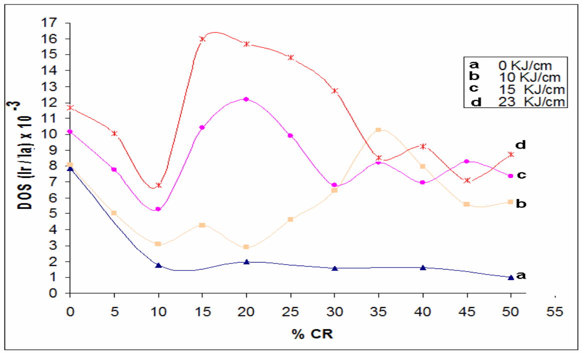

Figure 4: Effect of cold reduction on DOS (Ir/Ia) for various heat inputs

It is to be emphasized that such behavior appears to be not related to the introduction of ά- martensite since at 10% CR a negligible amount of this phase is formed. Moreover, the content of ά- phase is found to continuously increase with %CR. Meanwhile, such increase does not affect the DOS in a way that specimen's cold rolled in excess of 10% CR exhibit almost the same DOS even though they posses different contents of martensite phase. On the other hand the formation of martensite does not occur in the expense of carbides since transformation of carbides to martensite is not possible.

The above observation is supported by the microstructures of the cold rolled samples obtained either by the oxalic acid test or after EPR test. No evidence of IG ditches has been detected irrespective to the severity of deformation. The obtained results are in line with the results obtained by Mauzumi et al [8] who found that the corrosion rate of the solution annealed 304SS specimens decreased by a half as a result of 10% tensile straining, and further straining up to 30% had almost no effect.

As for curves b, c, and d correspond to welded samples shown in Figure (4), it is convenient to divide them into three distinct zones:

- The first zone (initial zone) which includes primarily cold work from 0% CR to 10% CR
- The second zone (intermediate zone) which extends from 10% CR to about 35% CR.
- The third zone (final zone) which covers cold reduction percentages from about 35% CR to 50% CR.

As for the first zone of Figure (4), the similarity between the trends of the curves of welded and unwelded samples may indicate the predominant role of the pre-existing corrosion attack in contributing in the total amount of IG corrosion.

It appears that the Ir/Ia values may not be due to the sensitization produced by welding alone. That is to say that although welding raises the DOS for all welded samples, but such increase is not enough to compensate for the large decrease in DOS caused by cold working the unwelded samples. Moreover, the increase in DOS of unworked (0%CR) welded specimens makes this compensation much more difficult. However, if the influence of initial CW on the Ir/Ia values of SS weldments is to be evaluated, the steel needs to be effectively solution treated. In this case, no sensitization will be left before subsequent welding that may falsify the end results. The behavior of 304SS in the intermediate range of Figure (4) is different. Where as increasing %CR value from 10% to 20% causes little effect on DOS for 10kJ/cm heat input, a large increase in DOS is noticed for 15 and 23 kJ/cm heat inputs. With increasing the % CR from 20% CR to about 35 % CR, the specimens show an adverse behavior. That is to say that, The DOS at 15 kJ/cm and 23 kJ/cm is observed to continuously decrease with % CR. Meanwhile, at 10kJ/cm heat input, it shows a gradual increase.

The sharp increase in the Ir/Ia ratio using 15 and 23 kJ/cm as a result of increasing %CR from 10 to 20%CR may be in part due to the high peak temperature accompanies such high heat inputs. The peak temperature seems to be high enough to suppress the cold worked structure in the HAZ up to approximately 20%CR. This in turn leads to higher localized attack at austenite grain boundaries. As a consequence of such removal of cold worked structure at the HAZ, the peak temperature would become the predominant parameter that governs the IG attack. Higher heat input implies higher peak temperature which in turn results in longer residence time at the sensitization temperature range so that the amount of IG corrosion is enhanced. This is not the case when welding with 10 kJ/cm heat input. The peak temperature appears to be insufficient to promote considerable sensitization. This is probably due to the fast rate of cooling experienced by the relatively thick specimens at such low levels of CW.

The role of CW is thought to be of importance at the onset of 20% CR. At such %CR, it is found that the original deformations appears to be large enough that can not be fully eliminated by either of the heat inputs used. The above observation may be supported by the non-disappearance of slip lines and deformation twins from the HAZ microstructure of specimen's cold rolled in excess of 20%CR for all heat inputs used. Increasing CW beyond about 20% CR leads to a corresponding increase in the non-recoverable dislocation density. Such increase seems to have a beneficial effect on reducing the DOS against the harmful influence of higher heat inputs (15, 23 kJ/cm).

At 10 kJ/cm heat input, an adverse influence is noticed. Although a considerable amount of cold worked structure persists at such lower heat inputs however, its influence may be diminished at lower peak temperature. Thus the effect of welding peak temperature on sensitization of cold worked material needs to be viewed in three ways:

- Its influence on the residence time at sensitization temperature range.
- Its influence on the removal of sensitization decelerating effect of prior working.
- Its influence on the diffusion mechanism of carbon and chromium.

Increasing CW results in decreasing the workpiece thickness which in turn reduces the cooling rate. Therefore at a given heat input, the residence time at sensitization range would be longer. Hence, from workpiece geometry viewpoint increasing %CR appears to have a harmful effect on DOS. On the other hand, increasing CW is reported by some authors; see for example [9, 10] to make the rediffusion of Cr to the depleted zone and homogenization of Cr much faster. Therefore the width of the depleted zone and the extent of Cr depletion could be less; hence the contribution to EPR value would be less.

Accordingly, for a given heat input and as a result of cold work, two contradicting effects on DOS may be recognized:

- A harmful effect due to extending the residence time at the sensitization temperature range.
- A beneficial effect owing to enhancing the Cr diffusivity to replenish into the depleted zones.

From the above observation, it may be suggested that the effect of cold work on IGC is well-related to the maximum temperature reached during welding. Even two opposite effects could exist in a way that increasing the metallurgical defects with CW appears to have a beneficial role in reducing the sensitization of 304SS weldments provided that the peak temperature is capable of enhancing the overall diffusivity of Cr as in the case of welding with higher heat inputs (15.23 kJ/cm). However, such a role of CW diminishes if the peak temperature is insufficient to cause such enhancement in Cr diffusivity as in the case of welding with 10kJ/cm.

The effect of cold reduction and welding heat input on the DOS of 304SS in the range of 35%CR to 50% CR (last region of Figure (4)) is less pronounced. This implies that increasing % CR in the above indicated range has little influence on Ir/Ia ratio independent of welding heat input. The less dependence of Ir/Ia on either %CR or heat input may be in part due to the following reasons:

- There appears to be a minimum amount of dislocation density required to cause partial replenishment of Cr to certain depleted zone irrespective of the amount of heat input. This critical amount is thought to be produced at approximately 35%CR. This implies that the compatibility between the two effects of cold work and peak temperature noticed at the intermediate degrees of CW seems to be no longer exist. Once the steel structure attains such minimum level of dislocation density, both effects of CR% and heat input on Ir/Ia values are found to be insignificant.
- This behavior may perhaps be related to the full penetration of weld observed at such thinner specimens. Full penetration may effectively raises the cooling rate because of surface radiation of heat. This in turn results in relatively rapid cooling and less heat efficiency which diminishes the role of heat input in determine the extent of intergranular attack of such specimens. Consequently, comparable values of Ir/Ia are found in this range of %CR. Meanwhile, full penetration indicates that much of the thermal energy is expended in melting the material instead of heating it which again minimizes the role of heat input.

From the above analysis, it seems that once full penetration is attained corresponding to a given thickness and heat input, a definite cooling rate exists at which the DOS is less influenced by heat input. Brümmer et al [11] found that a cooling rate of 10C˚/s did not cause appreciable chance in the DOS of 304SS (0.06 C) tested at different temperatures. Moreover, they found extreme variation in DOS at 1 C˚/s and 0.1 C˚/s. It is to be mentioned that the relative independence of DOS (Ir/Ia) on either cold work or welding heat input at relatively high levels of cold reductions is thought to be a unique observation of the current work. This behavior is rarely reported in earlier investigations, rather some change in DOS with CW and / or treating temperature is what mostly reported. The microstructural investigation obtained either by oxalic acid test or EPR test identify the sites where IG corrosion occurs. Most results indicate that sensitization takes place at austenite grain boundaries, and less likely at the ferrite/austenite interface of weld metal in some specimens. However, transgranular attack at austenite bulk matrix along the defects is rarely observed. The above observation is inconsistent with some other workers; see for example [12, 13] who reported the susceptibility of cold worked SS to transgranular attack when being sensitized. The difference may perhaps due to the limited time at which the material is at the sensitization temperature range due to welding compared to that encountered in conventional sensitization heat treatments.

The evolution of the EPR-microstructures of 304SS at different conditions is shown in Figure (5 from a to f). As it can be seen, the corrosion attack (weld decay) appears to be limited for the material at the area just beside the base metal in the HAZ (location d). This implies that this narrow zone of HAZ must have been exposed to temperature in the sensitization range a period of time that is sufficient to promote a considerable IG attack. The microstructures of Figure (5) are typical of most welded samples with some deviations that may exist depending on the locations from which the microstructures are taken.

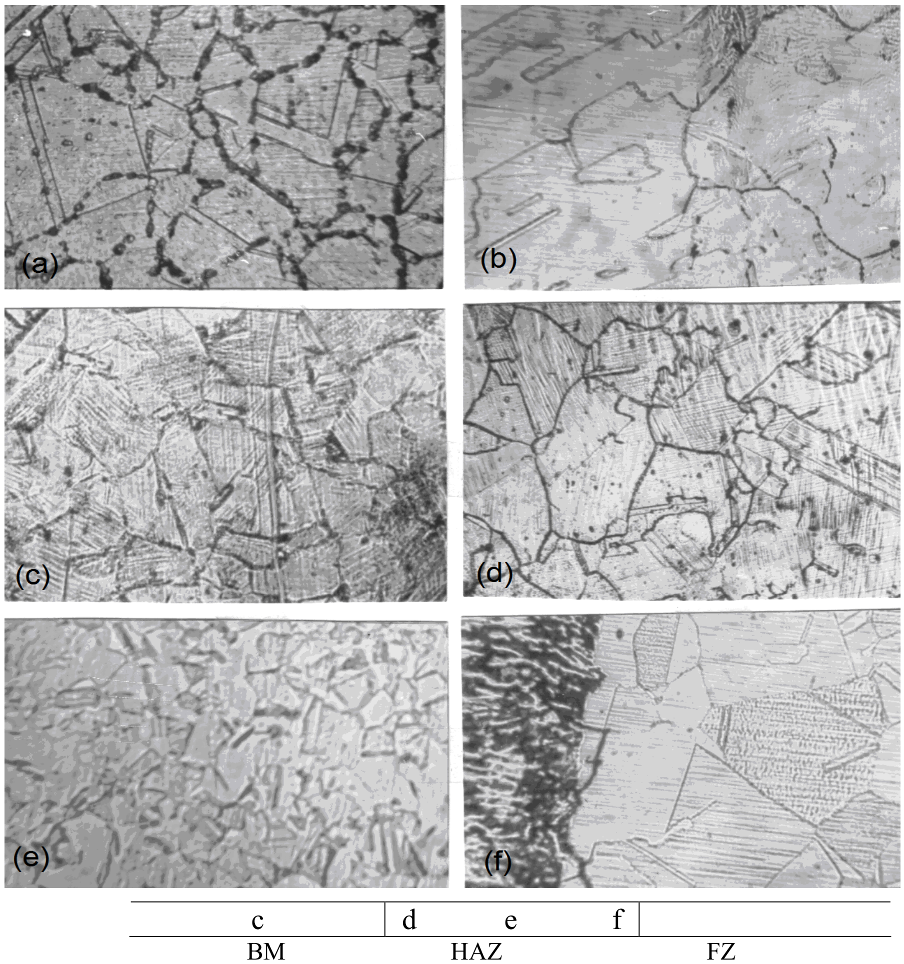

Figure 5: Microstructures of 304SS as (a) as received; (b) solution annealed; (c-f) across the weld of 20%CR specimen: (c) cold worked (base metal); (d) HAZ (weld decay); (e) recrystallization; and (f) weld interface, (Magnification 200x)

It is worthwhile to indicate that scanning electron microscope (SEM) is used to assess the existence of IG attack observed by the light microscope. In this case the attack is revealed as shallow grooves at the grain boundaries, see Figure (6).

(a)

(b)

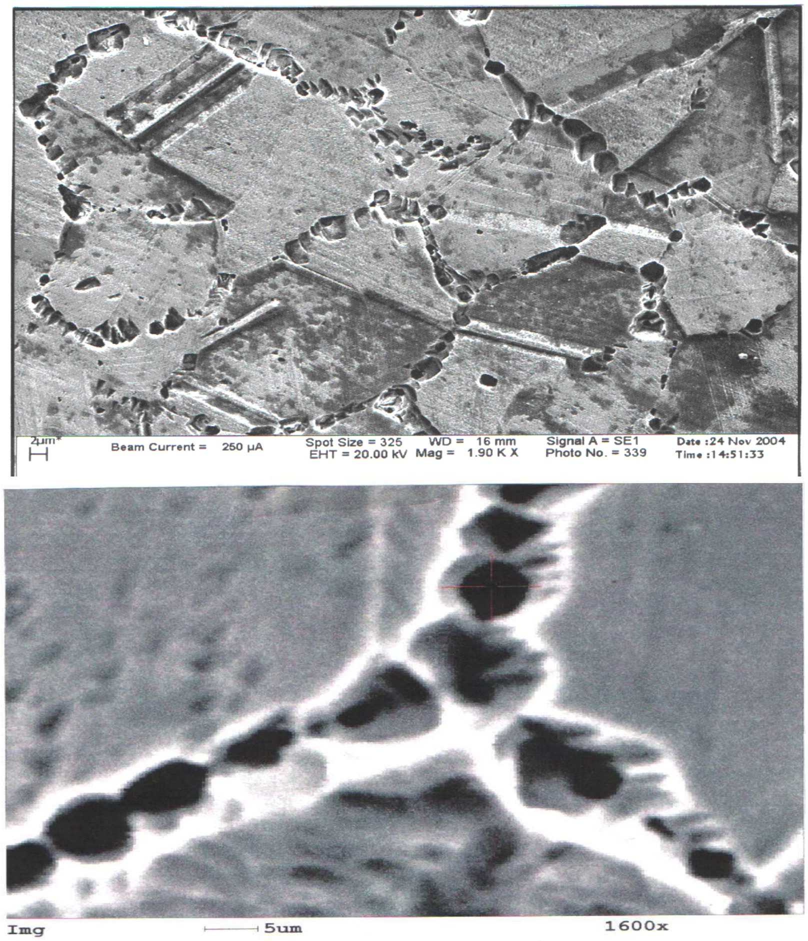

Figure 6: SEM micrograph of as received 304SS (a) continuous boundary grooves; (b) isolated ditches.

CONCLUSIONS

Based on the results obtained in this study the following conclusion can be drawn:

- The degree of sensitization as measured in terms of Ir/Ia ratio of unwelded solution annealed 304SS is shown to be extensively reduced when initially cold reduced to about 10%CR. Further deformation up to 50%CR does not cause appreciable change in the DOS.
- The results demonstrate that the influence of cold work on the DOS of 304SS welded specimens is highly recognized in the range of 10 to 35%CR. There appears

to be threshold values of CW beyond which the IG susceptibility of such specimens start decreasing. These values of CW are found to be inversely related to the welding heat input per unit length in a way that, 35, 20, and 15%CR are found to be the threshold values corresponding to 10, 15 and 23 kJ/cm heat inputs respectively.

- The DOS is found to increase with increasing heat input up to about 35%CR. At relatively higher levels of cold work ≥ 35%CR, the DOS appears to be less dependent on heat inputs. The results indicate that the DOS of almost pre-cold worked (≥ 35%CR) welded samples are comparable (Ir/Ia = 7 ±2) and being less than those of 0%CR welded specimens.

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