Research Article

Effect of Corrosion on Thermal diffusivity and Microstructure in the Weldment of Austenitic Stainless Steel AISI 310 in Acid medium

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Abstract

The effects of corrosion by 1M H₂SO₄ on the values of thermal diffusivity (α) in the weldment of austenitic stainless steel AISI 310 are studied. The samples were exposed for 48 hours in the corrosive solution at the room temperature. The 'a'values were measured by Photoacoustic spectroscopic (PAS) technique. The ' α ' value decreases in the welded and corroded welded samples. The decrease in the value of ' α ' shows the activity-passivity behavior in the corrosion process. Microstructural studies by Scanning electron microscopy (SEM) on the samples revealed the appearance of the irregular surfaces with the corroded grains leading to pits and porosity as a consequence of corrosion. The Energy dispersive analysis of X-ray (EDAX) shows the loss of chromium in the corroded welded specimen is more compared to the welded specimen. This may be due to the exposure in the corrosive H_2SO_4 solution, in which the sulfur is the effective promoter for the loss of Cr with the acid dissolution.

Keywords: Photoacoustics, Weld, Thermal diffusivity, Thermal conductivity, microstructure

This result shows the variation in thermal diffusivity which will be helpful for estimate the stability of weldments in acid environment.



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Introduction

Austenitic stainless steel (ASS) possesses excellent properties which range from high tensile strength, good impact resistance, corrosion and wear resistances have found various applications in many engineering industries. This material is used in almost all environments that require an optimization of these properties, some of which are low and high pressure boilers and vessels, fossil-fired power plant, flue gas desulphurization equipment, evaporator tubing, super heater reheating tubing and steam headers and pipes[1-3].

Welding is one of the main fabrication processes employed in almost all industries. It is basically a fusion of two or more pieces of metals by the application of heat and sometimes pressure [4]. The weldments are an indispensable part of most component fabrication, the frequent occurrence of weld failures has ensured ongoing research interest in the past few decades. The systematic investigations have been initiated to study the influence of variation in alloy microstructure in acid corrosion of stainless steel weldments, with a view to understand the thermal properties and environment which result in the weld failure. One of such failures is the corrosion cracking of a grade 304 stainless steel pipe improperly seam welded and meant for the conveying of glucose solution in Illinois USA[5]. The effect of

welding parameters reported on the corrosion of austenitic stainless steel in chloride medium [6]. Many other failures have proved to be welding prone or propagated. It is therefore relevant to investigate the influence of the thermal diffusivity of welded component in the corrosion behaviour of ASS in acid environments.

The thermal diffusivity is an important physical parameter that determines the heat transport through the material. This may be the essential condition of the proper choice of material and subsequently of the reliability and durability of the material. Since the thermal diffusivity is a parameter which depends closely on the microstructural variations, composition and the processing condition of the sample [7], it should be studied precisely. The thermal diffusivity of Fe-Co-Al alloys [8] is also reported by using this PAS technique.

Therefore, the main objective of this study is to determine the thermal diffusivity by photoacoustic technique at room temperature in the weldments of ASS immersed in one molar H_2SO_4 solution. The results obtained from this investigation are expected to provide more knowledge on the influence on the corrosion behaviour of ASS and the thermal cycle undergone by these welded structures during and after fabrication. This among other factors would help to estimate future disasters which may have occurred from failed parts due to corrosion of welded ASS structures. The microstructural analysis of the weldments was done by SEM and the elemental composition by EDAX.

Experimental

Sulphuric acid solution was prepared by mixing 60ml of concentric sulphuric acid (AR) in 1000ml of double distilled water. The AISI 310 stainless steel specimens were welded by metal arc welding with mild steel (MS3.15) as the filler material. The weld test specimens were cut in to an overall apparent size of 5 X 1 cm and were put in the acid (H_2SO_4) medium for 48 hours.

In the present photoacoustic spectroscopy experiment, a 450 W Xeon-lamp (Horiba Jobin Yvon, USA) is used as the source. The sample is placed in photoacoustic cell and the mike is placed very close to the sample. To get the modulated light, a mechanical chopper (C-995, Tetrahertz technologies Inc., USA) is used with the source. The photoacouctic (PA) signal from the microphone is fed to a lock-in amplifier. (SR-830 DSP Stanford Research, USA). The light is allowed to fall on the sample through a monochromator (Triax 180, Horiba Jobin Yvon, USA) as shown in Figure 1. The whole setup and the photoacoustic spectrometer is indigenously integrated and automated with the PC for the fastest data acquisition and better accuracy [9].



Figure 1 Schematic diagram of the photoacoustic spectrometer

For the present study, an open photoacoustic cell (OPC) is employed in the heat transmission configuration. Here the sample of size required is very small areas of around 2.3-1.5 mm ² is used. The open photoacoustic theory was developed [10] and later modified [11] by McQueen. In the OPC configuration, the solid sample is mounted directly on the top of the electret microphone leaving a small volume of air gap between the sample and microphone. When the sample is irradiated with chopped beam of polychromatic light, absorption of intermittent radiation and subsequent non-radiative de-excitation process is converted into heat. The pressure fluctuations produced in the gas which surrounded in the sample and the microphone inside the OPC cell generates the voltage in its output. This voltage is fed to the lock-in amplifier to record the signal amplitude and phase with respect to the chopping reference of the light incident.

The expression for the photoacoustic signal from the 1-D heat flow model of [12] is obtained as

$$\delta P = \frac{\gamma P_o I_o \left(\alpha_g \alpha_s\right)^{1/2}}{2\pi l_g T_o k_s f \sinh(l_s \sigma_s)} \exp\left[j\left(\omega t - \frac{\pi}{2}\right)\right] \tag{1}$$

Where, γ is the air specific heat ratio, P_o and T_o are the ambient pressure and temperature, respectively, I_o is the absorbed light intensity, f is the modulation frequency, l_i , k_i and α_i are the length, thermal conductivity and thermal diffusivity of the sample respectively. Here i = s subscript denotes the sample and i = g denotes the gas medium.

Also,
$$\sigma_s = (1+j)a_s$$
, where, $a_s = \left(\frac{\pi f}{\alpha_s}\right)^{\frac{1}{2}}$ is the thermal diffusion coefficient of the sample.

If the sample is optically opaque and thermally thick, then equation (1) reduces

$$\delta P = \frac{\gamma P_o I_0 (\alpha_g \alpha_s)^{1/2}}{\pi l_g T_o k_s} \frac{\exp \left[l_s (\pi f / \alpha_s)^{1/2}\right]}{f} \times \exp \left[j \left(\omega t - \frac{\pi}{2} - l_s \alpha_s\right)\right]$$
(2)

According to Equation (2), for thermally thick sample, the phase of the PA signal varies with modulation frequency 'f'. Hence, thermal diffusivity can be obtained from the phase data.

Results and Discussion

PA measurements: PA depth profile



Figure 2 PA spectrum

This is by keeping wavelength fixed, the chopping frequency is varied and the corresponding PA signals are observed. For the estimation of thermal properties of samples, depth profile analysis is sufficient. Thermal wave signals are amplified into many folds even a minute crack or defects on the surface detected by depth profiling. These

cracks could not be able to detect ultrasonic or metallographic technique. By the following general experimental procedure, PA spectrum has been obtained and that is shown in Figure 2. The thermal diffusivity ' α_s ' can be obtained either from the amplitude data or from the phase data (PA depth profile analysis) by exponentially fitting it to the amplitude data or linearly fitting it to the phase data respectively. The phase of the PA signal for different modulation frequencies is noted from the lock-in amplifier.

Knowing the coefficient 'a' from the fitting procedure ' α_s ' is readily obtained from, where $\mathbf{a}_s = \left(\frac{\pi f}{\sigma_s}\right)^{\frac{1}{2}}$ is the sample

thickness.

Similarly, the governing equation for the calculation of thermal conductivity, $k = \alpha \rho c_p$ Where, is the thermal diffusivity, p' is the mass density and $r_{cp'}$ is the specific heat capacity of the sample at constant pressure.

Thermal diffusivity decreases in the weld and corroded weld sample compared to the as received sample because more corrosion damages due to the microstructural variation in the weld zone. In the corroded weld specimen, the depletion of chromium by the corrosion induced rate will be more leading to the damages in the passive film. The acoustic waves are basically phonons, and these phonons will be very sensitive to the structural changes. Presence of grain boundary mismatches, defects in the passive film, etc. are expected to alter mean free path of phonons and this will be pronounced in the thermal diffusivity. Hence it is expected that the corroded weld sample has less thermal diffusivity than as received. There is no appreciable variation in the thermal diffusivity of the weld sample compared to the corroded weld sample. The thermal diffusivity of the as received sample in our measurement as shown in Table-1 is in good agreement with the value reported earlier [13].

Sample	Thickness (mm)	Thermal diffusivity (x ⁵⁻ 10 m ² /sec)	The rmal conductivity (m ⁻¹ K ⁻¹)
As received	1.8	0.41	15.89
Weld	2.0	0.20	7.75
Corroded weld	2.0	0.09	3.49

Table 1 Thermal diffusivity and conductivity of the samples

The welded and corroded weld sample exhibit much lower value of thermal diffusivity compared to as received sample. The lower value of thermal diffusivity can be attributed to the activity-passivity behavior on corrosion process [14]. Micro structural study by SEM revealed the appearance of irregular surfaces with corroded grain frontiers, porosity and fractures of micrometer size as a consequence of corrosion. It has been found that the lower value of thermal diffusivity in the weld sample was predominantly due to high temperature oxidation and thermal stress played the major role during the welding process. In the corroded weld sample, the depletion of the chromium will be more leading to the deterioration of passive oxide film. It may be due to intergranular corrosion (IGC). Sulfur is an effective promoter of IGC in these materials at both oxidizing potentials and those associated with active dissolution [15]. It should be noted that the welded joints in stainless steel may be serious problem in a structure due to possible damages due to corrosion.

SEM and EDAX

Microstructural study by SEM revealed the appearance of irregular surfaces with corroded grain frontiers, porosity and fractures of micrometer size as a consequence of corrosion. The SEM image of the weld without corrosion is shown in Fig.3. The SEM image of corroded weld in Figure 4 indicates the pit produced by the corrosive environment. The lack of fusion, the pinholes in the weld region and may provide a direct path for the corrosive

medium entering through it, leading to the formation of pits. The pits are irregular in shape and some of them are larger in size indicating the magnitude of corrosion product, which was confirmed in the EDAX.



Figure 3 SEM presentation of Weld region



Figure 4 SEM presentation of pitting damages in the corroded weld

From the EDAX spectrum by the Fig.5 and Fig.6, the higher content of carbon and lower content of chromium in the corroded weld sample as listed in Table.2 were probably the main reason for the corrosion. The traces of sulphur present in it also indicate the reason for the chromium depletion, which results in the change in thermal diffusivity value

The chromium (Cr) weight percentage was reduced in the corroded weld part as compared to the value of weld region which is tabulated in Table.2. It is clearly revealed the amount of chromium decrement was the poor corrosion resistance of weld corroded region.



Figure 5 EDAX analysis of weld region



Figure 6 EDAX analysis of corroded weld

Elements	Weld (%Weight)	Corroded weld (%Weight)
С	8.96	25.32
0	3.62	25.65
Si	0.76	1.6
Cr	14.63	6.37
Fe	60.96	31.46
Ni	11.08	4.49
S		4.28

 Table 2 Elemental composition (wt %) of the sample from EDAX

Conclusions

The PA analysis was successfully employed for the determination of thermal diffusivity of weld and corroded-weld samples under acid environment. The change in the thermal diffusivity value of the samples was reported in terms of activity-passivity behavior on corrosion induced failures leading to the breakdown of the passive film. The microstructural examination by SEM revealed the appearance of the pits and the EDAX shows the presence of sulphur which induces the change in the thermal diffusivity value in the corroded weld sample. Using EDAX analysis, it was shown that the austenitic stainless steel had different chemical composition than the nominal composition as a result of corrosion. A lower content of chromium (6.37%) and a higher content of carbon (25.32%) were found in the corroded weld samples than declared. The weld analyzed in this paper showed the tendency towards contact corrosion in the acid medium due to the inadequate welding process. The corrosion was the result of intergranular due to precipitation. The PA analysis of the weldment results, a prediction of whether the material could be in service without failures can be made, which will be helpful the life time estimate of the samples in near future.

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