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Localized interstitial element analysis in defect regions of a near alpha titanium alloy fusion weld

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This study was conducted on a fusion-welded specimen of Ti–6Al–2Nb–1Ta–0.8Mo to identify the cause of extensive cracking and porosity observed in the weld. Localized interstitial element measurements by nuclear reaction analysis techniques established that various regions in the weld and the base metal contained undesirably high concentrations of oxygen, carbon and hydrogen. A 5 μm thick surface layer of the as-received base metal, found to be rich in oxygen (16 000 w.p.p.m.), carbon (2700 w.p.p.m.), and hydrogen (425 w.p.p.m.), could have led to the multitude of weld defects observed in this work. In the weld and heat-affected zone, the hydrogen and carbon concentrations near various defects, such as cracks and porosity, were considered normal (i.e. $\sim$ 50 w.p.p.m. and 160 w.p.p.m., respectively). There were high oxygen concentrations ($\sim$ 3000 w.p.p.m.) near weld defects, which were two to three times higher than unwelded base metal. A direct correlation between oxygen content and microhardness showed that crack defect regions were characterized by both high oxygen and hardness, thereby indicating that oxygen played a major role in causing embrittlement and subsequent cracking.

1. Introduction

In titanium alloys the presence of oxygen, hydrogen and carbon in concentrations above acceptable limits can lead to cracking and porosity during welding [1–4]. Therefore, the possibility of localized interstitial element contamination, such as an oxidized weld bead surface, is of particular concern because it could seriously degrade mechanical properties and yet may not be detectable by bulk analytical techniques. This apparent insensitivity could occur because a relatively small volume of highly contaminated material located within a large, uncontaminated volume would not significantly increase the concentration of the large volume. This problem was encountered in recent work, where the formation of microcracks and voids in fusion welded Ti–6Al–2Nb–1Ta–0.8Mo (Ti-6211) was considered highly anomalous in view of the good weldability of the alloy [5]. Although interstitial element contamination was suspected as the probable cause of the defects, unusually high concentrations of these elements were not detected by inert-gas fusion techniques nor fluorescent X-ray spectroscopy. This led to an investigation to determine oxygen, hydrogen and carbon concentrations in localized regions near microcracks and weld pores in Ti-6211 welds, using nuclear reaction analysis techniques. The purpose of this paper is to present the results of localized interstitial element measurements in weld defect regions together with correlative microstructures and fractographic observations.

2. Experimental techniques

The material used in this work was cut from rolled and mill-annealed Ti-6211 plate. The multipass weld specimen analysed in this investigation was produced using the gas–metal–arc welding process with Ti-6211 filler wire in an argon protective environment. The bulk chemical analysis of base metal, weld metal and filler wire is shown in Table I. Samples of unwelded base metal representing both the as-received plate surface and internal bulk regions were included for subsequent nuclear probe analysis. Specimens were also cut from the weldment to expose internal weld defects and crack fracture surfaces, as shown in Fig. 1. The surfaces of the defect specimens selected for analysis, with the exception of fracture surfaces, were metallographically prepared by grinding to no. 600 grit silicon carbide and then final polishing with 0.05 μm alumina. After polishing, the specimens were not etched but were ultrasonically cleaned in acetone prior to microprobe analysis.

Microstructural and fractographic observations were made using optical and scanning electron microscopy (SEM). Knoop microhardness measurements (25 g load) were made on selected regions of polished and etched samples to evaluate variations within the weldment. Conventional inert-gas fusion and fluorescent X-ray spectroscopy techniques were used to determine bulk chemical compositions.

Hydrogen measurements were made on the weldment and the base metal surfaces using a nuclear reaction analysis technique [6, 7]. The technique is based on the detection of characteristic gamma rays emitted by a nuclear resonant reaction of a high energy lithium beam from a Van de Graaff accelerator with the hydrogen in the sample. The gamma-ray yield

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TABLE I Chemical analyses of Ti-6211 samples

Sample description	Analytical technique	Chemical composition								
		p.p.m. wt %				wt %				
		O	C	H	N	Al	Nb	Ta	Mo	Fe
Base metal–internal	Inert gas fusion; X-ray fluorescence	970 ± 112	300 ± 30	55 ± 2	85 ± 25	5.3	2†	1†	0.83	0.09
	Nuclear reaction analysis	1 175 ± 130	160 ± 10	65 ± 15						
Base metal–as-received surface	Inert gas fusion*	1 380 ± 105		65 ± 7						
	Nuclear reaction analysis	15 800 ± 1240	2700 ± 80	425 ± 44						
Weld metal	Inert gas fusion; X-ray fluorescence	1 210 ± 115	300	40 ± 3	130 ± 10	5.5	2	1	0.95	0.09
Filler wire	Inert gas fusion; X-ray fluorescence	855 ± 100	165 ± 35	20 ± 5	105 ± 5	5.8	2	1	1.3	0.04

*Fusion analyses of surface material were conducted on cut surface coupons = 0.050 in. thick.

† Not accurately determined because of insufficient calibration standards.

is directly proportional to the hydrogen concentration, and the calibration of the detection system is made using specially prepared surface standards [8]. All samples were hydrogen-probed using a 2.5 mm × 2.5 mm beam spot size. With the exception of the as-received base metal, the samples were probed at a single depth of approximately 1.8 μm below the exposed surface. The surface of the as-received base metal specimen was probed to depths of approximately 1.8, 2.6 and 3.4 μm. For the particular experimental conditions in this work, the minimum detectable limit for hydrogen was approximately 35 p.p.m. wt.

Oxygen and carbon were measured simultaneously using another nuclear probe technique [9]. In this technique, the surface region of interest is bombarded with deuterons that react with oxygen and carbon to produce a spectral distribution of emitted protons corresponding to the concentration of each element.

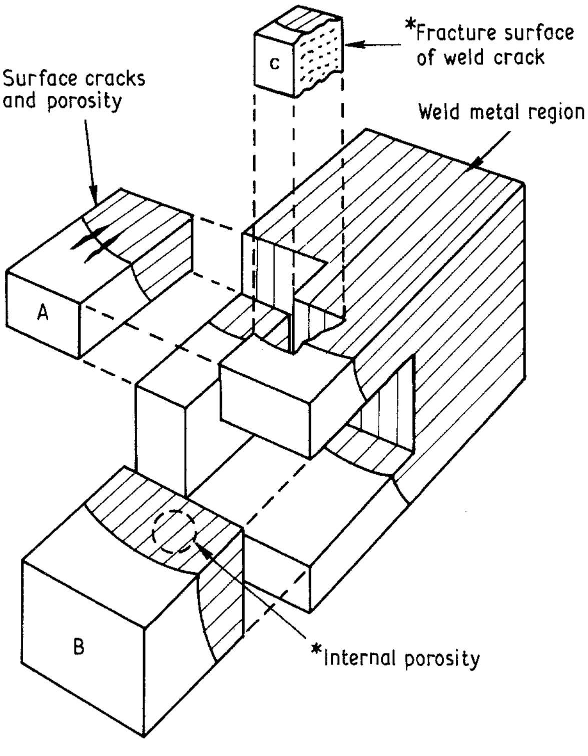

Figure 1 Schematic illustration of the location of specimens selected for nuclear reaction analyses. *Indicates surface analysed.

All samples were probed for oxygen and carbon using a 75 μm × 75 μm beam spot size. The data for each region probed represent the average oxygen or carbon concentration of a 3 μm thick layer, starting 0.25 μm below the surface. The first 0.25 μm surface layer was eliminated from the spectral analysis to avoid possible spurious surface film effects. In these experiments, the minimum detectable limits of oxygen and carbon were 300 and 20 p.p.m. wt, respectively.

3. Results and discussion

Radiographic and ultrasonic inspection of the weldment revealed the presence of internal flaws including pores, cracks and lack-of-fusion defects. The majority of pores were spherical and ranged from a few micrometres up to 2.5 mm diameter; their inside surfaces were silvery in colour. Intermittent groups of string-like porosity formed consistently in the weld metal near the fusion line of each weld pass. The cracks were also intermittent but were more widely spaced than the pores. Surface cracks were detected only in the outer weld pass, but in the same general location as the porosity. The cracks appeared to start in the weld metal and propagate toward the heat affected zone (HAZ). The crack path through the HAZ was typically jagged, with considerable crack branching and secondary cracking.

Typical chemical analyses for the base metal by conventional bulk fusion and nuclear probe techniques are given in Table I. Of particular interest are the unusually high concentrations of oxygen, carbon and hydrogen in the as-received base metal surface as measured by the nuclear probes. These results show that a hydrogen gradient existed at the surface, which decreased rapidly within a few micrometres, as shown in Fig. 2. On the other hand, the interstitial element analyses of Ti-6211 base metal specimens, which represented both surface and near surface material, failed to show the actual extent of surface contamination. This would be expected because the average concentration of a bulk specimen will be determined mainly by the larger uncontaminated volume rather than a thin, highly contaminated layer. For example, a 50 μm thick oxidized layer containing about 10 000 w.p.p.m. oxygen on a titanium specimen might

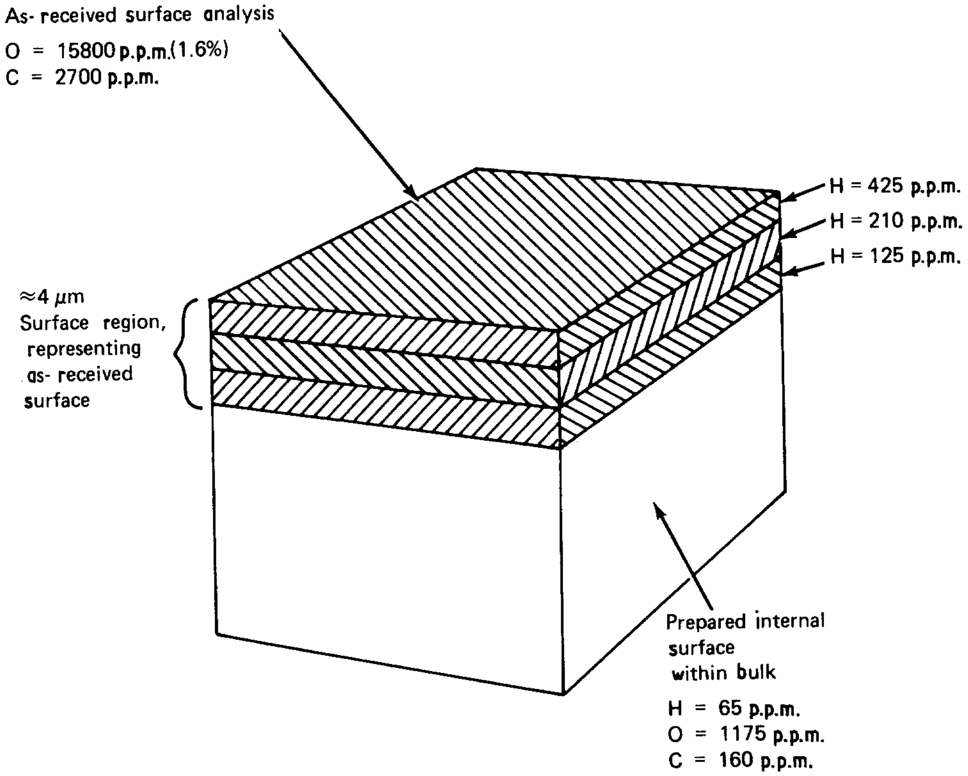

Figure 2 Analyses of base metal as-received and internal surfaces.

constitute only about 2% of the total mass. Thus, if the oxygen concentration in the uncontaminated region were 1000 w.p.p.m., the average bulk analysis of oxygen in the specimen indicated by the inert-gas fusion method would be approximately 1200 w.p.p.m. Such an analytical result would not at all indicate potentially harmful interstitial concentrations which, if not removed prior to welding, could result in weld defects.

Typical results of nuclear probe analyses for hydrogen in the proximity of a crack are shown in Fig. 3. The hydrogen concentrations around the crack in both the weld metal and HAZ are approximately the same as normal bulk values (i.e. ~ 50 w.p.p.m.). In fact, the hydrogen concentrations in the weld metal appeared to be somewhat lower than base metal values, which may be the result of outgassing during welding or because a low hydrogen filler wire was used. This trend was also indicated by bulk hydrogen measurements (Table I) and thus indicates that there were no unusual concentrations of residual hydrogen associated with weld cracks or pores after welding.

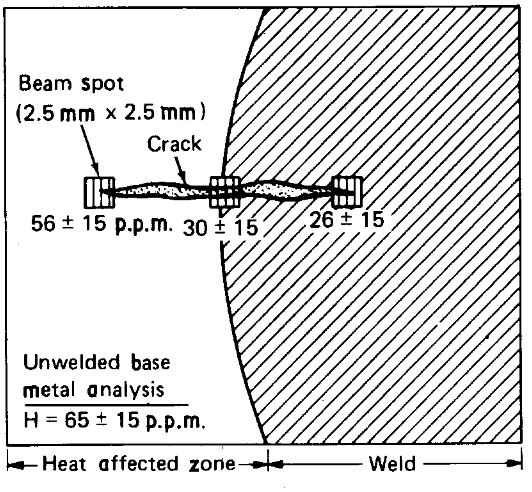

Figure 3 Hydrogen analysis on top weld surface – Sample A. Reliable detectability limit for hydrogen in these experiments was ~ 35 p.p.m.

In general, the results of analyses by the nuclear probe for oxygen technique showed definite oxygen enrichment in the weld samples, particularly near defects, as shown in Figs 4 and 5. The average oxygen concentrations tend to be higher at points within 0.5 mm of a crack or pore, both in the weld metal or HAZ. Peak oxygen concentrations in these areas were approximately 3000 w.p.p.m., or about two to three times that in the base metal and the HAZ. The high oxygen contents in the HAZ are attributed to diffusion during and after welding. The nuclear probe analysis showed that carbon concentrations within the weld and HAZ remained at low bulk levels (i.e. 160 w.p.p.m.). Although oxygen concentrations were high in these areas, it was not possible to identify the cause of the observed weld porosity in these samples (i.e. due either to the degassing of hydrogen or oxygen). It has been shown in other work that excessive oxygen (i.e. 1.0 wt %) could account for weld porosity [1, 10], but it is also well known that hydrogen can cause the smooth, shiny porosity observed extensively in these weld samples [1–4, 10–12].

The association between interstitial contamination and defect formation is further demonstrated by the results of oxygen and carbon analyses of a crack fracture surface representing both weld metal and HAZ, shown in Fig. 6. The weld metal surface was characterized mainly by cleavage fracture with some ductile tear ridges, high concentrations of oxygen and carbon, and a dark oxide-like discolouration. This suggests that interstitial contamination and welding

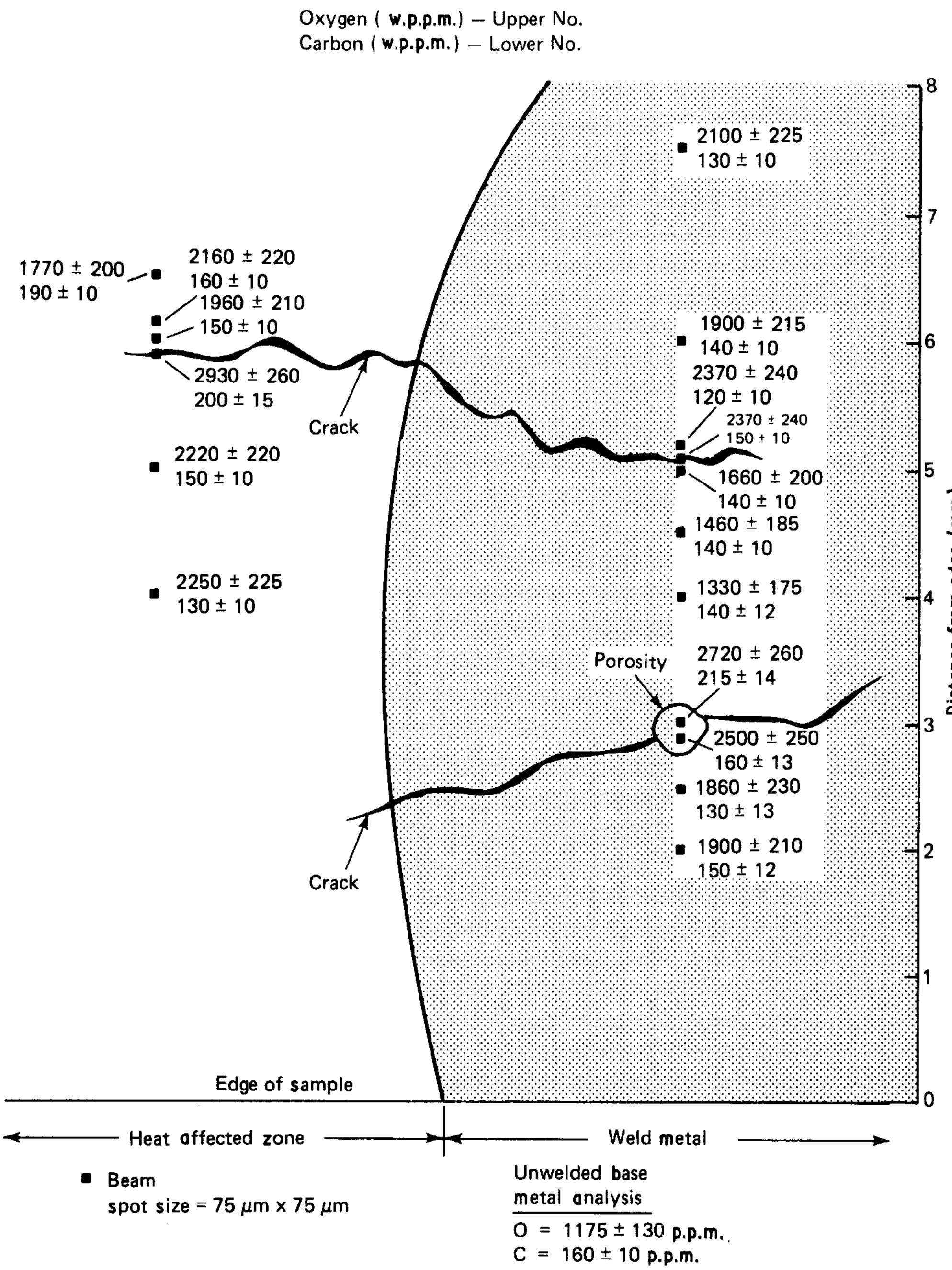

Figure 4 Oxygen/carbon analysis on top weld surface – Sample A.

restraint stresses in the relatively hot weld metal led to the formation of cracks which subsequently discoloured due to oxidation and eventually propagated to the cooler HAZ. The fracture surface of the HAZ was characterized by a ductile, dimple rupture mode, relatively low oxygen and carbon concentrations, and no oxide discolouration.

The results of microhardness tests show that significantly higher hardnesses were associated with cracks in both weld metal and HAZ. A summary of the average interstitial concentrations and the corresponding average microhardness for selected regions near cracks is given in Table II and schematically shown in Fig. 7. The hardness data represent the material in the general

TABLE II Average microhardnesses and interstitial element concentrations in various weldment regions

Region analysed	Knoop microhardness (25 g load)	Nuclear reaction analysis (w.p.p.m.)		
		Oxygen	Carbon	Hydrogen
Base metal – unwelded	255 ± 40	1175 ± 175	160 ± 10	65 ± 15
Heat affected zone (> 0.5 mm from crack)	285 ± 65	2020 ± 215	165 ± 10	–
Heat affected zone (< 0.5 mm from crack)	345 ± 100	2350 ± 420	170 ± 10	55 ± 15
Weld metal (> 0.5 mm from crack)	280 ± 50	1700 ± 275	140 ± 10	–
Weld metal (< 0.5 mm from crack)	355 ± 75	2325 ± 355	170 ± 10	35 ± 15

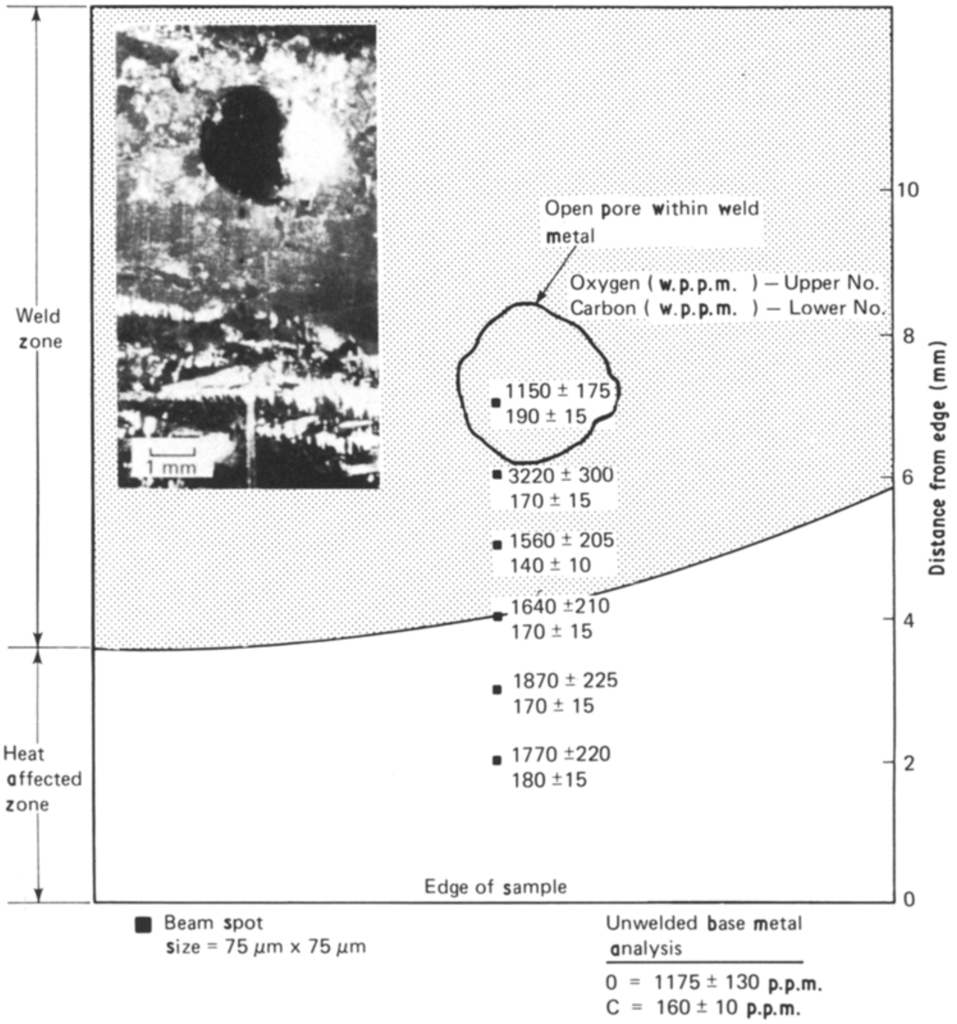

Figure 5 Oxygen/carbon analysis near internal weld porosity – Sample B.

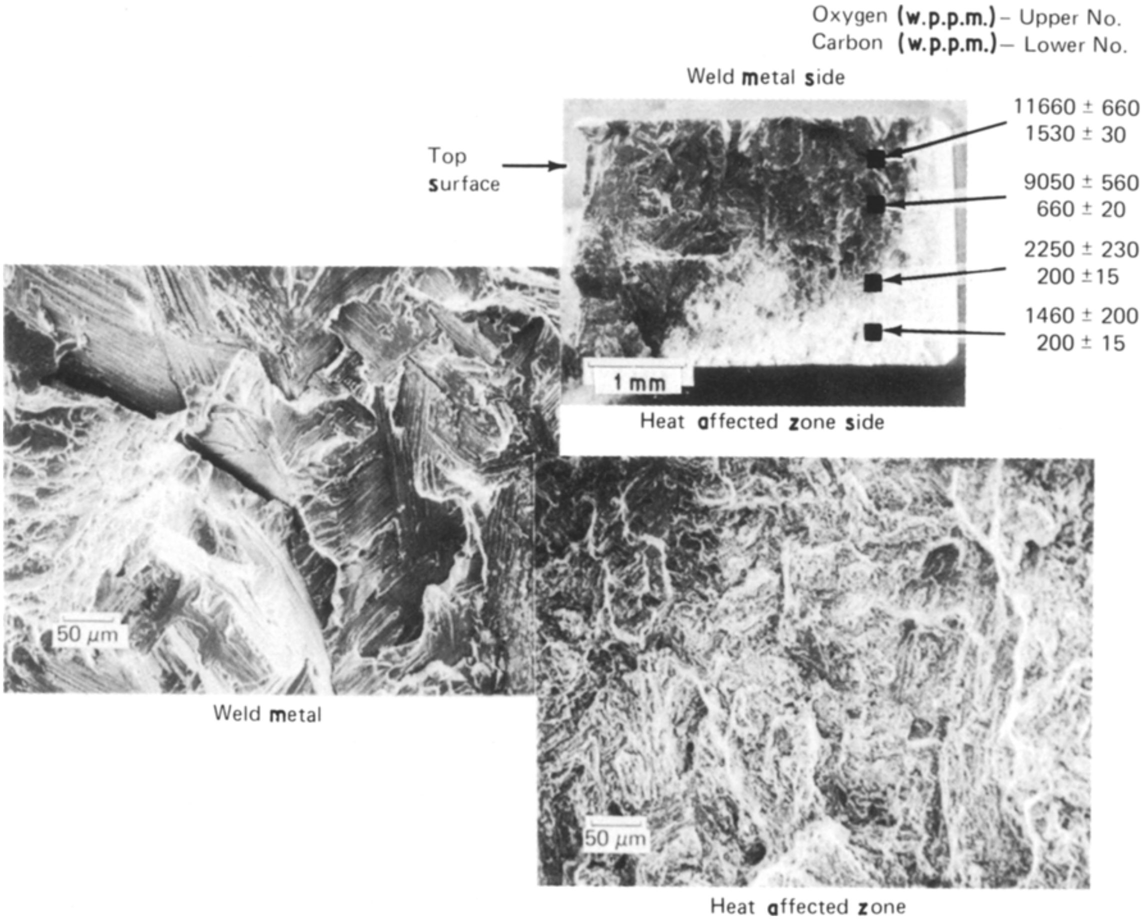

Figure 6 Oxygen/carbon analyses of crack fracture surface – Sample C. SEM fractographs.

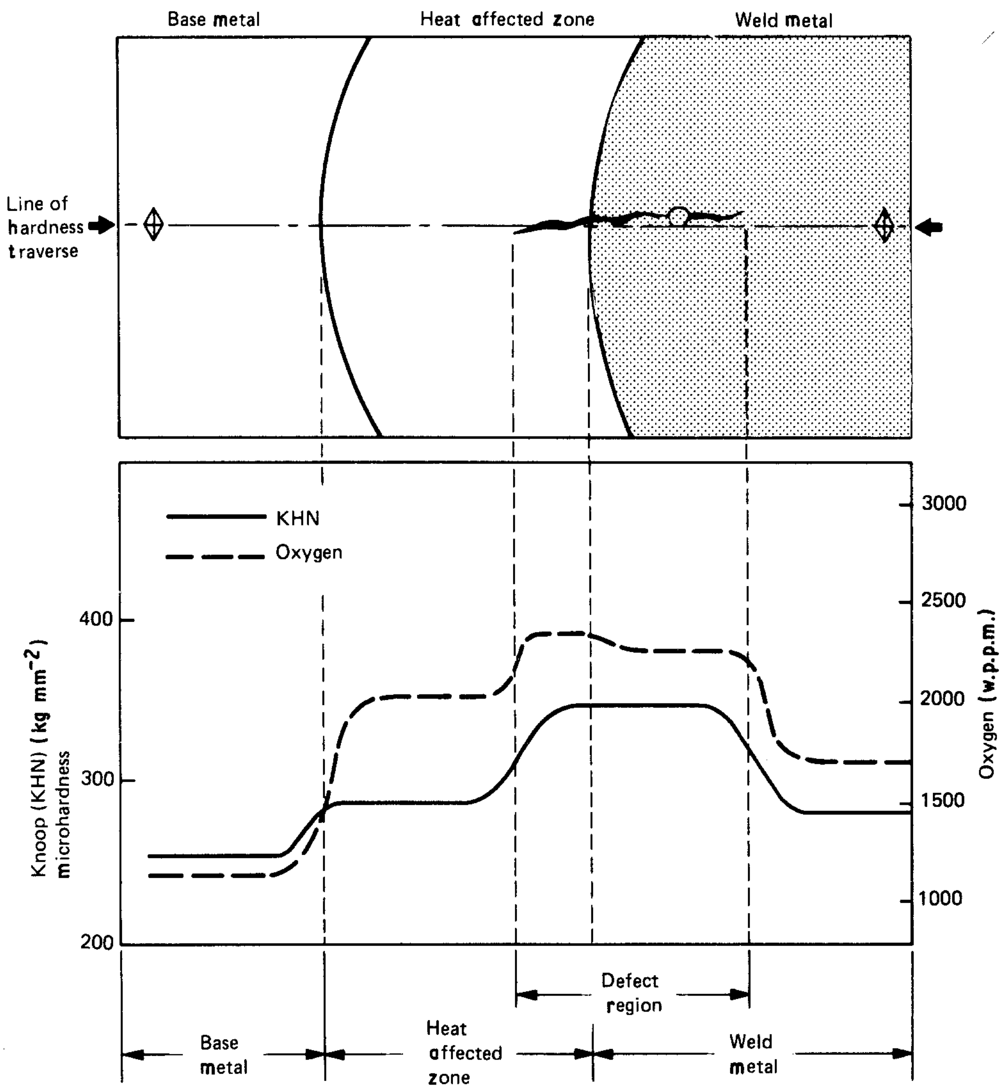

Figure 7 Schematic representation of average oxygen content and microhardness across Ti-6211 samples.

vicinity of the oxygen measurements but not necessarily the same spot measured by the nuclear probe. In general, the average oxygen concentrations and hardnesses in very close proximity to weld cracks were higher than those of surrounding material. Crack regions with an oxygen concentration of approximately 2300 w.p.p.m. corresponded to a microhardness of 350 KHN. The carbon and hydrogen concentrations in these same regions were of the same order as the bulk values. Based on the observed variations in hardness and oxygen content within the weld, it appears that regions which were oxygen enriched and relatively harder were susceptible to crack formation.

4. Conclusions

1. The Ti–6Al–2Nb–1Ta–0.8Mo titanium alloy weld was characterized by abnormally high oxygen concentrations near weld cracks, indicating that oxygen played a major role in causing embrittlement and subsequent cracking.

2. The as-received base metal surface was highly contaminated by oxygen, carbon and hydrogen. Failure to adequately remove such a surface layer before welding could produce the kind of weld defects observed in this investigation.

3. Since the bulk hydrogen concentrations in the weld and HAZ were normal, there were no direct data to indicate that hydrogen caused the extensive weld porosity that was observed.

4. The nuclear probe techniques used for interstitial analysis are very effective for quantitative measurement of oxygen, carbon and hydrogen concentrations in thin surface layers and at localized defects in materials.

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