To the Graduate Council:

I am submitting herewith a thesis written by Harsha Bhojraj entitled "Effects of Shielding Gas Purity and Gas Purification on the Behavior of Gas Metal Arc and Gas Tungsten Arc Welds". I have examined the final copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science in Metallurgical Engineering.

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EFFECTS OF SHIELDING GAS PURITY AND GAS PURIFICATION ON THE BEHAVIOR OF GAS METAL ARC AND GAS TUNGSTEN ARC WELDS

A Thesis

Presented for the

Master of Science

Degree

The University of Tennessee, Knoxville

Harsha Bhojraj

May 1995

DEDICATION

This thesis is dedicated to my parents

Vijaya Bhojraj

and

B.T. Bhojraj

ACKNOWLEDGEMENTS

I am indebted to my major professor Dr. C.D. Lundin, for providing the opportunity and instruction to successfully execute this work. The guidance received will continue to be positively instrumental in all my future work.

I am grateful to my parents, B.T. Bhojraj and Vijaya Bhojraj, who left no stone unturned to provide their children with good education and values. I am similarly grateful to my brother Naveen Bhojraj who has guided me in all my major endeavors, especially this one at UTK.

I also wish to thank my committee members, Dr. R.A. Buchanan, and Dr. C.R. Brooks, whom I regard as exemplary instructors.

I would also like to express my appreciation for my hard working colleagues and members of the welding group, Dr. K.K. Khan, Dr. C.Y.P. Qiao, Liu Peng, Wang, Karen, Zhou, Henry, and Kameel. Thanks are also due to the support staff in the MSE department, Mike Neal, Frank Holliway, Sandy, and Mrs. Cagle.

I would also like to thank Dr. Paul Bhadha, a sponsor member of this project for his useful contribution and friendly encouragement. Likewise, I also wish to thank chief sponsor Ken Krysiak for his valuable support throughout the program.

I also thank my fiance, Nathalie Rouhana, for her ceaseless support especially during the tough times. Finally, I am grateful for the many solid friendships that have been forged during this period as a graduate student and I wish to express my appreciation for all who have made my stay here pleasant and worthwhile.

ABSTRACT

The work presented investigated the effects of shield gas purity and gas purification on the behavior of gas metal arc welds and gas tungsten arc welds of important industrial alloys. A "Nanochem" resin-based purification system was used to purify the inert argon and helium shielding gases used . The effect of moisture (up to 300 ppm) and oxygen (up to 100 ppm) contamination deliberately introduced into the shield gas was investigated, and the subsequent benefit derivable from the use of "Nanochem" purification was assessed. The feasibility and effectiveness of using a newly patented design concept (Gas Injection) to prevent shield gas contamination in gas delivery systems of GMA and GTA torches was also evaluated.

Nanochem gas purification when GMA welding aluminum 1100, 5556, and 6061 with shielding gas originally contaminated with moisture and oxygen resulted in significant improvements in weld surface appearance. In contrast, the surface appearance of GMA welds in 5086, 6061, and 1100 aluminum was significantly deteriorated when welding with contaminated gas. Similar findings were observed in the GTA welding of aluminum 1100. Nanochem purification also significantly reduced porosity in GMA welds of 5086 and 6061 aluminum, resulting in significant improvements in bend ductility. Corrosion properties were also significantly improved in Nanochem purified welds as determined by electrochemical polarization tests. However, in aggressive corrosive mediums, the beneficial effect of purification was lost.

In autogenous GTA welds on AL-6XN alloy, Nanochem purification consistently and significantly offset the deleterious oxidation effects created by

moisture and oxygen contaminated argon shield gas. Heat tinting was also totally eliminated. Corrosion resistance properties, as determined in various acidic and caustic media, although not quantitatively significant, were qualitatively better in Nanochem purified welds.

In GTA weld deposits of 100-S high strength steel filler wire, Nanochem purification effectively reduced the diffusible hydrogen level by up to 20 ppm, when welding with moisture saturated gas lines. When the oxygen and moisture impurities were added to the shield stream, Nanochem purification reduced the diffusible hydrogen levels by 2 ml/100g.

In autogenous GTA welds made on aluminum 1100, 316 and 304 stainless steel, titanium, and 255 duplex stainless steel, Nanochem purification was effective in preventing gross weld pool contamination and deleterious oxidation of as-welded surfaces caused by shield gas impurities.

In the GMA welding of alloys such as HY-80 steel and 304 stainless steel, the deployment of Nanochem was ruled out because of its incompatibility with the necessary active shielding gas component (2% oxygen). Attempts to weld with inert gas devoid of any active components was deemed inappropriate due to degradation of arc characteristics and bead appearances. Similar, but not so serious effects were found in the GMA welding of duplex 2205 stainless steel using argon, which lacked the 2% recommended oxygen additive.

GMA welded 2205 weldments qualitatively exhibited a higher corrosion resistance when shielded with argon-2% oxygen without Nanochem. Toughness properties, however, were significantly degraded in the welds shielded with the recommended argon-2%oxygen shield. In contrast, Nanochem-purified, originally contaminated gas was found to exhibit the

highest toughness properties. The effect of moisture up to 300 ppm in the argon shield gas did not seem to degrade the toughness, bend ductility, and the corrosion behavior of the 2205 duplex stainless steel weldments.

No problems with the purity of the bottled gas cylinders were experienced throughout the entire work. Welds made with bottled gas were comparable to welds made with Nanochem purified gas. In field situations however, gas purity may not always be satisfactory due to cylinder-to-cylinder variations, manifold cross-contamination problems, mositure permeation into gas plumbing, and/or minute piping leaks. On the basis of the test results, the use of Nanochem is highly recommended in such situations to eliminate such problems that may cause weld contamination.

"Gas-injection" modification to prevent air entrainment in the automatic GTA weld torch resulted in regular shield stream disruption. Hence, it is not recommended for torches of this general configuration. In the GMA torch used in this study, a new concept called "dual-injection" was implemented in conjunction with "gas-injection" modifications to prevent air entrainment in the wire carrying conduit of the welding system.

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CHAPTER 1

RESEARCH MOTIVATION AND PURPOSE

The importance of shielding gas and its purity on the quality of a gas-shielded weld has always been common knowledge in the welding community. Welding grade shielding gases are usually 99.995% pure and fully satisfactory for fabrication purposes. Nevertheless, there always exists a possibility of shield gas contamination arising from minute piping leaks, rusted cylinders, cross-contamination in manifold systems, inadequacies in purging procedures, and leaky fittings. Any of these factors can significantly degrade the shielding gas purity at the weld pool. In order to combat the possibility of shielding gas contamination arising from different sources and to ensure consistent gas purity levels at the weld pool, Hercules, Inc., developed a "Nanochem" resin-based gas purification system. Studies conducted by Hercules using this purification system indicated a distinct improvement in the toughness properties of E-Brite and Ferralium alloys, which were gas tungsten arc welded with Nanochem-purified welding grade gas.

The potential sensed in these preliminary experiments with shielding gas purification provided the motivation for this present study. This study endeavored to further explore the possibility of improvements in weld metal properties of other alloy systems using Nanochem gas purification. Accordingly, several alloy systems were test welded with the GMA and GTA processes, using different shielding gas purity levels, with and without the use of Nanochem. These test welds were examined for the effects of shield gas

contamination and subsequent benefits derivable from Nanochem purification. The alloys employed in the study were: 304 stainless steel, 316 stainless steel, 255 duplex stainless steel, 2205 duplex stainless steel, AL-6XN alloy, titanium, HY-80 steel filler wire, 1100 aluminum, 5086 aluminum, and 6061 aluminum.

The candidate alloys were chosen with the calculated anticipation that they would be susceptible to the degrading effects of impurities. Moreover, they were also selected from the standpoint of their extensive use in industry. Depending on the alloy used, different properties were tested in the different welds in order to establish and compare the differences between welds shielded with purified gas and welds shielded with contaminated gas. Contaminants employed in this study were moisture and/or oxygen in amounts ranging from 10-300 ppm and 2-100 ppm, respectively. Although different tests were conducted on the different alloys, a common test always included was evaluation of the as-welded surface appearance. This simple but conclusive methodology was used because of its importance in estimating the overall integrity of a weld. Visual inspection is oftentimes the only test employed for process and component quality certification and hence, a degraded weld surface can often warrant component rejection at the fabrication stage itself. Other evaluations encompassed corrosion testing in different media for a qualitative insight into the corrosion behavior as a function of shielding gas purity. Bend testing and impact testing were the main mechanical properties examined. Metallography and hardness testing were also employed when necessary to evaluate pore defect levels and changes in the microstructural characteristics. The arc characteristics as affected by shielding gas were also subjectively investigated.

GTA and GMA torch modifications to incorporate "gas-injection" were also carried out. Gas-injection is a patented concept of Hercules, Inc., which attempts to prevent entrainment of air in the wire feed system of GMA and GTA welding equipment. The necessity and efficiency of this proposed concept was evaluated for benefits claimed in terms of improvement in arc characteristics and weld pool shielding protection.

CHAPTER 2

INTRODUCTION TO SHIELDING GASES

Since the inception of gas shielded arc welding, shielding gas research has been a continuing industry endeavor, testimony of which is seen in the multitude of shielding gas blends available today. A general overview of the popular shielding gases is presented in table1. The quest to develop new gases remains a continuing effort and gases are getting increasingly customized to meet specific applications. Researchers have regularly compiled information on available shielding gases [1, 2, 3, 4, 5, 6, 7, 8, 9, 10]. The suitability and interaction of different shielding gases with different alloys within different processes has also been regularly critiqued by researchers [11, 12, 13, 14, 15, 16, 17, 18, 19].

Figure 1 indicates the practically insignificant cost of shielding gas (3% of the total welding process costs). There can be few areas in metal fabrication where the choice of a low cost element has a more dramatic impact on the overall efficiency of production than in the selection of the shielding gas for welding operations. This concern has spurred the advancement of shielding gas technology to the extent that a common welding fabricator today can select the most optimum shielding gas by simply dialing into an easily accessible expert computer system. Such systems analyze the relevant details such as component parameters, process variables, weld properties and production costs to recommend the most suitable shielding gas blend for fabrication [20].

Table 1. Overview of Shielding Gases

SHIELDING GAS	GAS REACTION	GMAW AND FCAW	GTAW AND PAW
Pure gases			
Argon Ar	Inert	Nonferrous	All metals
Helium He	Inert	Nonferrous	Al, Mg, and Cu alloys
Carbon dioxide CO_2	Oxidizing	Mild and low alloy steels (some stainless steels)	Not used
Two component mixtures			
Argon mixtures			
Ar + 20-50% He	Inert	Al, Mg, and Cu alloys	Al, Mg, and Cu alloys
Ar + 1-2%CO_2	Oxidizing	Stainless and low alloy steels	Not used
Ar + 3-5% CO_2	Oxidizing	Mild, low alloy, stainless steels	Not used
Ar + 20-30% CO_2	Oxidizing	Mild, low alloy steels (some stainless steels)	Not used
Helium Mixtures			
He + 25% Ar	Inert	Al and Cu alloys	Al and Cu alloys
CO_2 mixtures			
CO_2 + upto 20% O_2	Oxidizing	Mild and low alloy steels	Not used
CO_2 + 3-10% O_2	Oxidizing	Mild and low alloy steels	Not used
Three component mixtures			
He + 75% Ar + 25% CO_2	Inert	Stainless and low alloy steels	Not used
Ar + 3-10% O_2 +15% CO_2	Oxidizing	Mild steels	Not used

Source: "Modern Welding Technology", Howard Cary-Prentice Hall

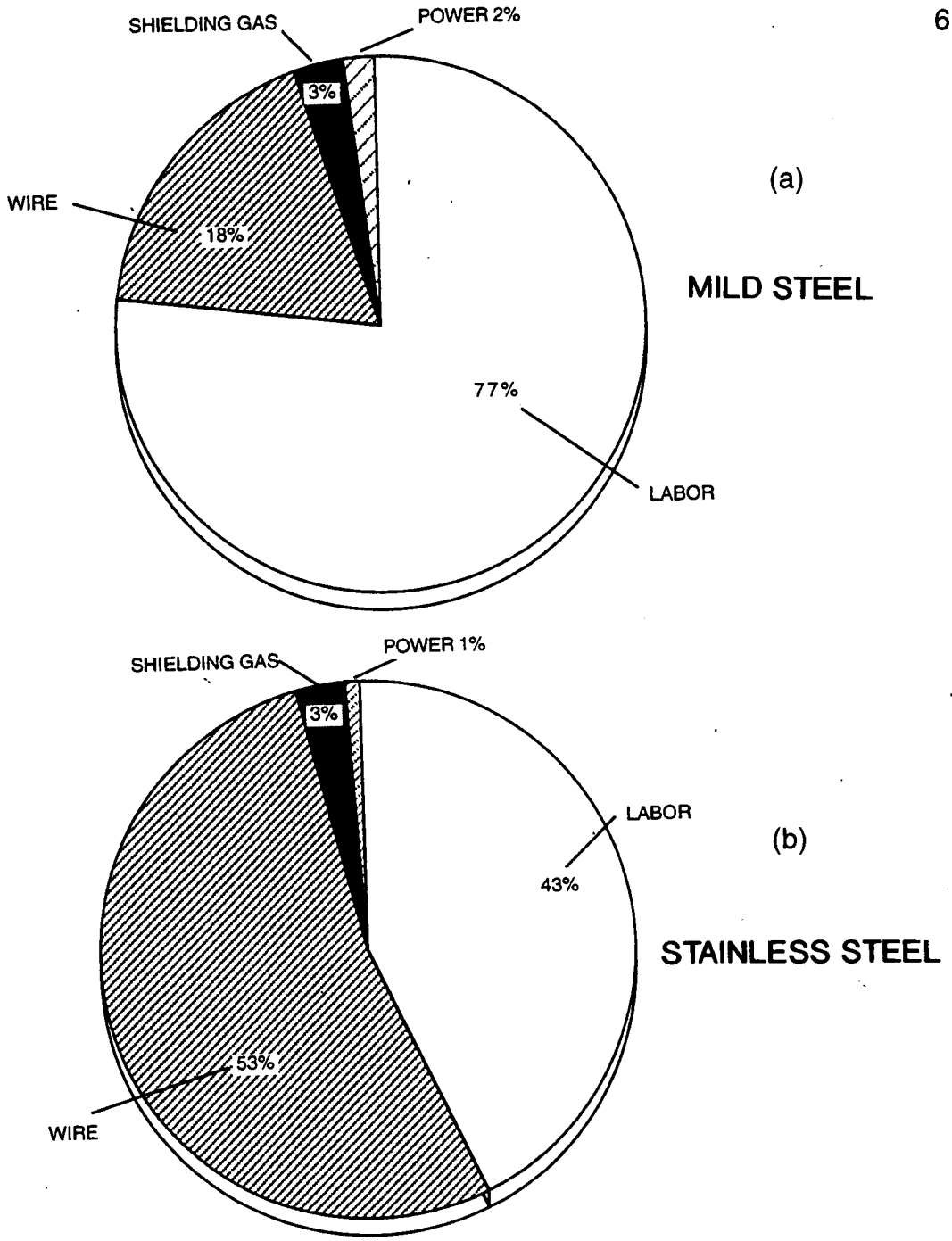


Figure 1. Relative cost of shielding gas per meter of weld.
(a) Mild steel (b) Stainless steel

Adapted from: "Shielding Gases for Gas Metal Arc Welding", D. Hilton, Welding and Metal Fabrication, July 1990

Shielding gas standards are constantly being updated and revised. In 1993, a new set of standards was made mandatory in the EEC (European Economic Community), covering titles such as shield gases, hose connections, and gas tightness of welding equipment [21].

The following intoductory sections address several aspects of shielding gases such as the history and development, manufacture, and properties of important shield gases and shield gas additives.

A Historical Perspective Of Shielding Gas

The history of shielding gas development [22] began in the late nineteenth century when Charles Lewis Coffin replaced the air in a box placed over a welding joint with a non-oxidizing atmosphere. The next forty years witnessed sporadic periods of interest in inert, non-oxidizing shielding gases. Then in 1930, two U.S. patents were issued that are considered to be the first descriptions of the use of inert shielding gases for welding. The first patent, issued to H.M. Hobart, described the use of helium with carbon or metallic arcs. The other, issued to P.K. Devers, described the use of argon and its mixtures for arc processes.

The Northrup Aircraft Company first used an inert gas shielded welding process in the early 1940's with a non-consumable welding electrode. The process was developed specifically for the welding of magnesium for aircraft fabrication using helium as the shielding gas. Sensing its potential, the Linde Division of Union Carbide Corporation acquired the invention in 1942 and initiated an extensive research and development program to expand the use of

the process. Introduced commercially in 1946 as the Heliarc process, it is known today as the Gas Tungsten Arc Welding (GTAW) and Tungsten Inert Gas (TIG) process.

A significant part of the Heliarc development work was devoted to the evaluation of argon and helium as shielding gases, and to studies that identified the importance of gas purity in producing quality welds. It was not until the postwar era, after Linde pioneered the economical production of high purity argon on a commercial scale, that Heliarc (GTAW) became a practical reality.

In 1950, a patent was issued to the Air Reduction Co., Inc., (Airco) covering a process later known as Gas Metal Arc Welding (GMAW). The initial application was spray arc welding of aluminum in a helium atmosphere. The process then was known as SIGMA (Shielded Inert Gas Metal Arc). Extensive development work was undertaken by Linde, Airco, and others during the 1950's. The effort was dedicated to developing gas mixtures, wire chemistry, and equipment systems to improve and expand the application range of the process. The rapid growth of the process thereafter is witnessed in the major use of the process today. The rapid growth also provided the groundwork for the invention of the Flux Cored Arc Welding process (FCAW) by Arthur Bernard in the late 1950's. His patent was assigned to the National Cylinder Gas Co. (NCG) in 1957, where the process was further developed and introduced for practical use. Also, during the 1950's, Linde developed, patented, and commercialized Plasma Arc Cutting and Welding Systems. The basic patents covering this discovery were issued in April 1957.

Gas shielded welding processes are now in worldwide use and play a major role in the fabrication of products for the aerospace, railroad, trucking, and construction industries, to name a few.

2.2. **Shielding Gas**

The primary function of the shielding gas is to exclude the atmosphere from contact with the molten weld metal. Most metals when heated in air to near their melting point will form oxides, and to a lesser extent, nitrides. Oxygen will also react with carbon in the molten steel to form carbon monoxide and carbon dioxide. These varied reaction products may result in weld deficiencies, such as trapped slag, porosity, and weld metal embrittlement. Reaction products are easily formed in the atmosphere unless precautions are taken to exclude nitrogen and oxygen. In addition to providing a protective environment, the shielding gas and flow rate also affect the following:

1. Arc characteristics
2. Mode of metal transfer
3. Penetration and weld bead profile
4. Speed of welding
5. Undercutting tendency
6. Cleaning action
7. Weld metal mechanical properties

2.3. **Shielding Gas Selection**

Selection of a shielding gas is made on the basis of how it will affect the following criteria:

1. Soundness and geometry of the weld bead
2. Suitability of the arc-voltage arc length characteristic for automatic control
3. Rapidity of arc ignition
4. Ease of puddle control (manual welding)
5. Cost of making the weld

Different gases and different gas additives have varied effects on each of the factors described. The specific characteristics of the common shield gases and gas additives, and the effects they have on the properties described above are addressed in the subsequent section.

2.4. **Gas Shielded Arc Welding Processes**

2.4.1. **Gas Tungsten Arc Welding**

The GTA process is illustrated in figure 2a. It uses a nonconsumable tungsten (or tungsten alloy) electrode held in a torch. Shielding gas is fed through the torch to protect the electrode, molten weld pool, and solidifying weld metal from contamination by the atmosphere. The electric arc is produced by the passage of current through the conductive, ionized shielding gas. The arc is established between the tip of the electrode and the work.

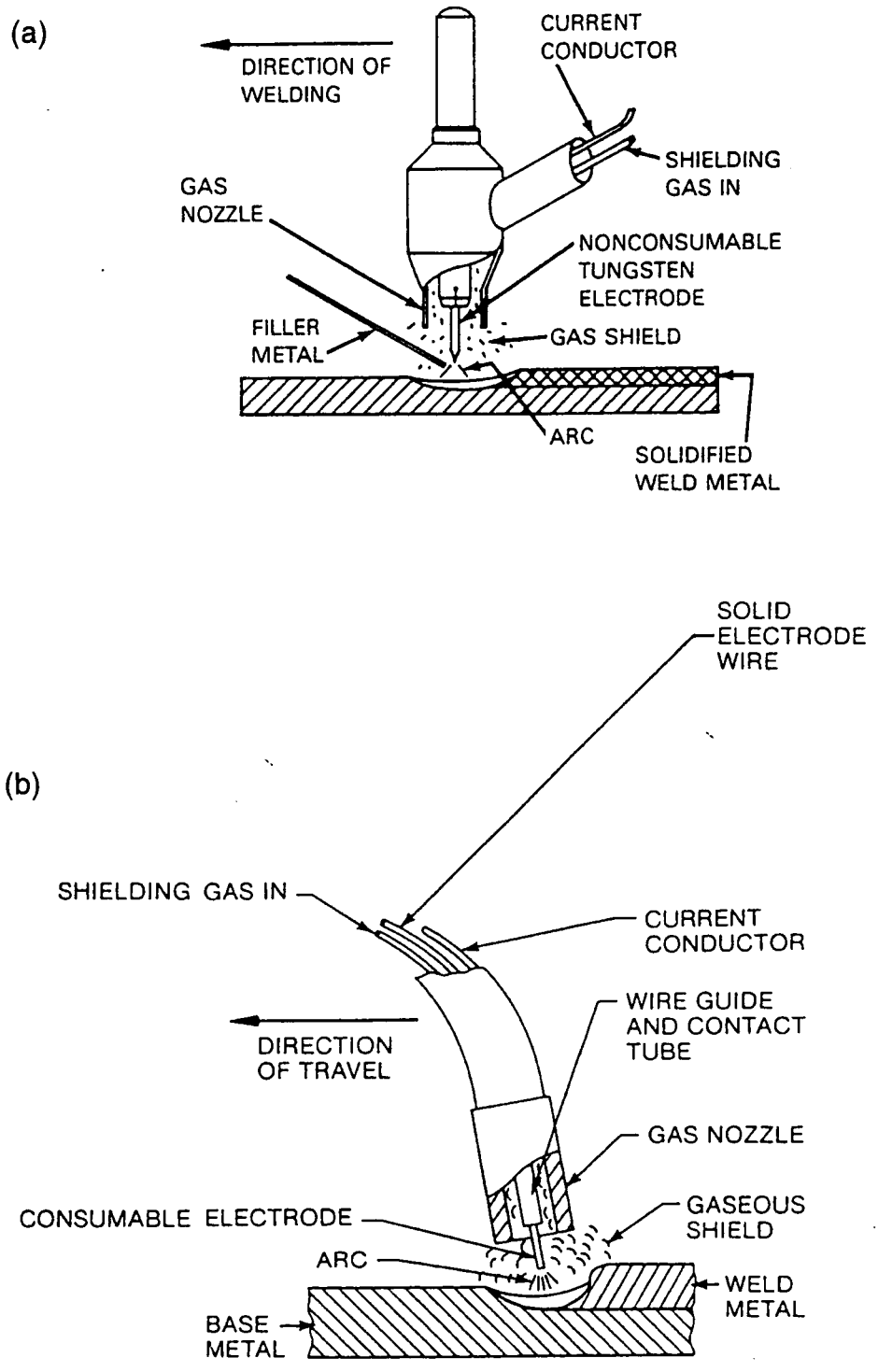


Figure 2. Gas shielded welding processes.
(a) Gas tungsten arc welding (b) Gas metal arc welding

Source: Welding Handbook, Vol. 2, 8th edition

Heat generated by the arc melts the base metal. Once the arc and weld pool are established, the torch is moved along the joint and the arc progressively melts the faying surfaces. Filler wire, if used, is usually added to the leading edge of the weld pool to fill the joint. Four basic components common to all GTAW setups as illustrated in figure 2, are the torch, electrode, power source, and the shielding gas.

Advantages:

1. It produces superior, defect-free quality welds.
2. It is free of spatter.
3. It can be used with and without filler metal.
4. It allows excellent control of root pass weld penetration.
5. It can produce inexpensive autogenous welds at high speeds.
6. It allows precise control of welding variables.

2.4.2. **Gas Metal Arc Welding**

GMAW is an arc welding process that uses an arc between a continuous filler metal electrode and the weld pool. The process is shielded by an externally supplied gas and without the application of any pressure. The process is illustrated in figure 2b. After initial settings by the operator, the equipment provides for automatic self-regulation of the electrical characteristics of the arc. Given proper equipment and settings, the arc length and the current (wire feed speed) are automatically maintained. When the welding torch is track mounted, even the travel speed and gun positioning are easily maintained.

Advantages

1. It is the only consumable electrode process that can be used to weld all commercial metals and alloys.
2. It overcomes the restriction of limited electrode length encountered with shielded metal arc welding.
3. Welding can be done in all positions, unlike submerged arc welding.
4. Deposition rates are significantly higher than in shielded metal arc welding.
5. Welding speeds are higher because of continuous electrode feed and higher filler metal deposition rates.
6. Because the filler wire feed is continuous, long welds can be deposited without stops and starts.
7. When spray transfer is used, deeper penetration is possible than with shielded metal arc welding, which may permit the use of smaller size fillet welds for equivalent strengths.
8. Minimal post-weld cleaning is required due to the absence of a heavy slag.

Physical Properties of Gases

Some general physical properties of welding shielding gases are presented in table 2. Five properties of welding shielding gases, however, deserve special mention as they have important effects on welding. The five important gas properties are: thermal conductivity, dissociation and recombination, reactivity, surface tension, and gas purity.

Table 2. Properties of Common Shielding Gases

	Argon	Helium	Carbon dioxide	Hydrogen	Nitrogen
Chemical symbol	Ar	He	CO_2	H_2	N_2
Atomic number	18	2	-	1	7
Molecular weight	39.95	4	44.1	2.016	28.01
Specific gravity, air=1	1.38	0.1368	1.53	0.0695	0.967
Density (lb/cu.ft) at 0^0C, 1 atmosphere	0.1114	0.0111	0.1235	0.0056	0.0782
Ionization potential (eV)	15.7	24.5	14.4	13.5	14.5
Thermal conductivity@ 32^0F (10^{-3}XBtu/hr-ft-^{0}F)	9.69	85.78	8.62	97.22	13.93
Cubic ft.lb.	9.67	96.71	8.73	192	13.8
Cubic ft./gal.	113.2	100.6	74.0	103.7	93.2

Source: Shielding Gas Selection Manual, Linde Publication

The relevance of the five important gas properties and their effects on welding are discussed in the subsequent sections.

2.5.1. **Thermal Conductivity**

The ability of a gas to conduct heat influences the radial heat loss from the center of the arc column to the periphery. Argon, which has a low thermal conductivity, produces an arc that has two zones: a narrow hot core and a considerably cooler outer zone. As a result, the penetration profile of the weld fusion area exhibits a narrow 'finger' at the root and a wider top. On the other hand, a gas which has a high thermal conductivity will conduct more of the heat outward from the core, resulting in a wider, hotter arc. This type of heat distribution, which occurs with helium, argon-hydrogen, and argon carbon-dioxide mixtures, gives a more even heat input to the work piece surface and produces an overall wider profile throughout the fusion zone. High thermal conductivity is essential when joining highly conductive metals such as aluminum and copper.

2.5.2. **Dissociation and Recombination**

Shielding gases such as carbon dioxide, hydrogen, and oxygen breakdown or dissociate into free radicals and ions when heated to the high temperatures of the arc plasma. They are then at least partially ionized, producing free electrons and current flow. As the free radicals come in contact with the relatively cool work surface, the atoms recombine and in the process

generate additional heat. This process does not occur with stable, monoatomic gases such as argon. Therefore, at the same arc temperature, the heat generated at the work surface can be greater with gases such as carbon dioxide, hydrogen, and oxygen.

2.5.3. **Reactivity**

Reactivity, as it applies to shielding gases, is a comparative measurement of how readily a shielding gas will react with the molten weld metal. Argon and helium are completely non-reactive, or inert, and therefore have no effect on the weld metal. Nitrogen is sometimes considered an inert gas. However, at the high temperatures associated with welding, it may react and have an effect on weld chemistry. Oxygen and carbon dioxide fall into a category of reactive gases known as oxidizers. These gases will react with molten metal in the arc to form oxides. Hydrogen falls into the category of reducing gases. Hydrogen will preferentially react with an oxidizing agent over the molten weld metal, thereby helping to prevent the formation of oxides in the molten weld metal. However, when used on hardened steel, hydrogen can produce some adverse effects, such as underbead cracking.

2.5.4. **Surface Tension**

In any liquid, there is an attractive force exerted by the molecules below the surface on those at the surface. An inward pull or internal pressure is thus created, which tends to restrain the liquid from flowing. Its strength varies with

the chemical nature of the liquid. In welding, the surface tension between the molten steel and the surrounding atmosphere has a pronounced influence on bead shape. If the energy value is high, a convex, irregular bead will result. Low values promote flatter beads with minimum susceptibility for undercutting. Pure argon shielding is usually associated with high interfacial energy, producing a sluggish puddle and a high crowned bead. These characteristics are partially attributed to the high surface tension of the liquid iron in an inert atmosphere. Iron oxides, however, have a considerably lower surface tension and thus promote good wetting to the parent metal. Therefore, in GMA welding, for example, the addition of small percentages of oxygen or carbon dioxide to argon results in a more fluid weld puddle.

2.5.5. **Gas Purity**

Although some metals, such as carbon steel and copper, have a relatively high tolerance for contaminants, others such as aluminum and magnesium, are fairly sensitive to impurities. Still others, such as titanium and zirconium, have extremely low tolerances for any foreign constituent in the shielding gas. The dew points and moisture contents of welding grade shielding gases are shown in table 3.

The issue of gas purity constitutes the main concern of this study and is addressed in complete detail in subsequent sections.

Table 3. Dew points and moisture concentrations of welding grade gases

	Minimum purity %	Maximum moisture ppm	Approximate dew point at maximum moisture content (^{0}F)
Argon	99.995	10	-77^0
Helium	99.95	32	-61^0
Carbon dioxide	99.5	34	-60^0
Hydrogen	99.95	8	-80^0
Nitrogen	99.7	32	-61^0

Note: Moisture specifications are measured at full cylinder pressure, the pressure at which the cylinder is analyzed

Source: Shielding Gas Selection Manual, Linde publication

2.6. **Common Gases and Gas Additives**

A myriad of gas mixtures are in common and varied use today, in a vast mosaic of applications worldwide. However, the basic shield gases and gas additives continue to be the following:

Argon

Helium

Carbon dioxide

Oxygen additive

Hydrogen additive

Nitrogen additive

Varied combinations of the above gases

The traits and characteristics of these gases are described below.

2.6.1. **Argon**

Argon is an inert monoatomic gas with a molecular weight of 40 g/mol and is obtained from the atmosphere by the liquefaction of air. Welding grade argon is refined to a minimum purity of 99.95%. For reactive and refractory metals, a minimum purity of 99.997% is required.

The use of argon has the following advantages to offer:

1. Smoother, quieter arc action.
2. Reduced penetration.
3. Better cleaning action when welding materials such as aluminum and magnesium.

4. Lower costs and greater availability.
5. Lower flow rates for good shielding.
6. Better cross draft resistance.
7. Easier arc starting.

Argon is 38% heavier than air, which makes it advantageous for welding in the flat position and the horizontal fillet position. Pure argon can be used as a shielding gas in the GTA welding of virtually all materials.

2.6.2. **Helium**

Helium is a light, chemically inert gas and is obtained from the separation of natural gas. Welding grade helium is refined to a purity of at least 99.99 percent. It is used primarily on aluminum, copper and magnesium alloys. Because it is lighter than air, high gas flow rates must be used to maintain adequate shielding. Also, because it floats up and maintains good shielding, helium is used favorably in the overhead welding position. Globular metal transfer is obtained with helium shielding, but spray transfer can also be obtained at high current levels. Hence, a larger degree of spatter and a poorer weld bead appearance are produced as compared to argon. For any given arc length and current level, helium will produce a hotter arc, which makes it good for welding thick sections and metals with high thermal conductivities. Helium generally produces wider weld beads and better penetration than argon.

2.6.3. **Carbon Dioxide and Carbon Dioxide Additions**

Carbon dioxide is a reactive gas commonly used in its pure form for GMA welding carbon and low alloy steels. It is the only reactive gas used alone as a shield in GMAW. Higher welding speed, greater joint penetration and lower costs are general characteristics that have encouraged the extensive use of CO_2 shielding gas. When welding with solid or flux cored wire, carbon dioxide is commonly used for shielding.

Due to the dissociation and recombination of its component parts, carbon dioxide has a higher thermal conductivity and transfers more heat into the metal. A broader penetration pattern is obtained, but base plate distortion and greater joint melt-through are possible problems. Because carbon dioxide is an active gas, more oxidation of the filler metal will occur during welding. This oxidation necessitates the use of a higher alloy welding wire in order to minimize the chances of weld porosity and to maintain necessary strength levels. This gas/wire mixture will often produce a weld bead with a tenacious layer of manganese or silicon oxide that must be removed prior to any further processes like painting. When pure carbon dioxide is used, a complex interaction of forces occurs as metal droplets form at the wire tip. These unbalanced forces cause large, unstable droplets to grow and transfer to the weld pool in a random manner. The addition of carbon dioxide provides the same beneficial results as the addition of oxygen. The dissociation of carbon dioxide enhances arc stability and bead wetting. Because it has less oxidizing power than oxygen, less slag is produced on the bead surface. As a consequence, less post-weld cleanup is required.

2.6.4. **Oxygen Additions**

Although oxygen itself is not used as a shielding medium, it is a vital component of many modern gas shielding mixtures. Oxygen appears to perform two functions. Firstly, it aids stability within the arc by ionizing readily within it (13.6 eV). Secondly, and more important, is the effect oxygen has on the surface tension and arc rooting of both the weld and the droplets of filler material. When GMAW is carried out in the free flight mode, additions of oxygen to the inert shield reduce the surface tension at the droplet and promote the formation of a stable arc root. These effects aid the formation of small droplets and axial transfer, which in turn reduces the amount of spatter and increases the wettability of the molten weld pool.

The weld bead crown is also flattened, reducing excessive weld reinforcement. The transition current in the GMAW process is substantially influenced by oxygen. As the percentage of oxygen in the shielding gas increases, spray transfer, with higher deposition rates and low fume and spatter levels, can be achieved over a wider range of operating parameters. The oxygen lowers the required transition current and expands the useful working envelope for the process.

However, excessive levels of oxygen can be detrimental to the process. Alloy losses start to increase. The amount and tenacity of the scale on the surface of the weld bead also increases and some deterioration in the weld mechanical properties occurs. Therefore, oxygen addition is limited to a maximum of 5%, which minimizes the oxygen-associated problems and at the

same time, also provides the desired operational benefits. It is relevant to note that when using the GTA process, the use of oxygen as a shield gas additive is strictly prohibited to prevent oxidation of the tungsten electrode.

2.6.5. **Hydrogen Additions**

Hydrogen may be used as a shielding gas or as a shielding gas additive because its chemical activity is of a reducing nature. For the same reason, it also has no adverse effects on the tungsten electrode in TIG welding. Hydrogen possesses an extremely high arc-voltage characteristic that is effective in direct proportion to its content in the gas mixture. Evidently, hydrogen is much more effective than helium as an additive to argon for producing higher voltages. The high arc voltage yields high arc power at relatively low current levels.

Hydrogen also burns on exposure to atmospheric air, providing an excellent shield at extremely low gas flows, generally about one-sixth of that required with helium. By virtue of the high voltage and low gas flows obtainable, argon-hydrogen mixtures, up to and including 35% hydrogen, are preferred to argon-helium mixtures for welding materials where the hydrogen does not cause adverse metallurgical effects (eg. stainless steel, nickel copper alloys, and nickel base materials). A 20% addition of hydrogen to argon produces arc voltages approximately equivalent to those of helium. Production experience has proved that these shielding gases perform equally well.

2.6.6. **Multiple Shielding Gas Mixtures**

Argon-oxygen carbon dioxide

Gas mixtures of argon with up to 20 percent carbon dioxide are versatile. They provide adequate shielding and desirable arc characteristics for spray, short circuiting, and pulse mode welding.

Argon-helium-carbon dioxide

Mixtures of argon, helium and carbon dioxide are used with short circuiting and pulse arc welding of carbon steel, low alloy steel, and stainless steels. Mixtures in which argon is the primary constituent are used for pulse arc welding. Mixtures in which helium is the primary constituent are used for short circuiting arc welding.

Argon-helium-carbon dioxide-oxygen

This mixture, known as the "Quad Mix", is popular for high deposition GMAW using the high current density metal transfer type arc. This mixture will give good mechanical properties and operability throughout a wide range of deposition rates. Its major application is in the welding of low alloy, high tensile base materials, but it has also been used on mild steel for high production welding. Weld economics are an important consideration in using this gas for mild steel welding.

2.7. Gas Flow and Shielding Efficiency Aspects

"How much gas is required to produce an acceptable weld?" This question constitutes an important issue in gas shielded arc welding. Given the

vast multitude of applications, processes and equipment, it is hard to prescribe specific flows during welding. However, several recommendations in the form of nomograms and data charts exist that aid in the selection of gases and optimum flow rates for the welding of different alloys [23].

Gas shielding requirements, mechanisms and coverage assessments have found some mention in the literature. Studies have addressed the factors affecting the flow of gas and principles that can aid the judgment of those concerned with using shielding gas in an efficient manner. Fedorenko and Shvedikov [24] used numerical methods to characterize the gas shielding zone in the GTA process and concluded that four distinct regions are associated with the formation of the gas shielding zone.

Gibson [25] introduced the concept of 'gas coverage' in spot welds to characterize the effectiveness of gas shielding. It is defined as the area on the surface of the workpiece from which air is excluded by the shielding gas. (The effect of air in the shield gas in terms of changes in gas coverage was quantified by the use of GTA spot tests in aluminum and titanium.) Other researchers have also developed different methods to evaluate shielding gas coverage effectiveness. The following methods have been described in literature [26]:

1. Schlieren tests
2. Shadowgraph tests
3. Heated coupon tests
4. Sputtering tests
5. Smoke tests
6. Weld and spot tests

In 1988, Awano, Kishi and Suzuki [27] developed a new visualization method for evaluating shielding characteristics that assists in studying the blowholes formed during welding. Air current tests are also an important aspect of gas coverage assessment because air drafts are often a practical problem to reckon with during gas metal arc welding. The influence of air movement on the flow characteristics of shielding gas has been examined by Bezbakh [28]. Khvorostov [29] has determined the optimum flow rates for the welding of 18-8 stainless steel in the open. Similarly, Autio and Strom [30] have discussed the harmful effects of air currents and their prevention in MIG welding. Tekriwal [31] has developed a mathematical model to assess the effect of torch angle and shielding gas flow on TIG welding.

Research aimed at improving shielding effectiveness is important and bound to continue because defects are often directly related to poor shielding efficiency.

CHAPTER 3

BACKGROUND

3.1 Sources of Shielding Gas Contamination

The two main inert shielding gases, argon and helium, can be and are refined to equivalent purity specifications. It is possible to refine both gases in welding grades to purities of 99.99% (superior grade) by prepurging the cylinders prior to filling. Most GTAW users specify welding grade gases of 99.995% purity. At this level of purity, differences in the kind and relative amount of impurities are not detectable in the welding operation. But at these specifications, the gas still contains 20-25 ppm of total impurities that may have an effect depending on the material being welded. Carbon steel and copper are apparently forgiving to even large amounts of contaminants. Magnesium and aluminum are sensitive to impurities present in the shield gas. Titanium and zirconium have extremely low tolerance for any foreign constituent in the inert gas. Hence, different metals respond differently to the contaminants present in the shield gas.

The principal factors responsible for shielding gas contamination are the following:

1. Pre-contaminated, rusty gas cylinders
2. High permeability flexible gas hoses
3. Damaged, defective or loose shielding/backing lines
4. Defects in the piping systems

5. Leaky fittings
6. Cross contamination in manifold systems
7. Poor purging practice

In view of the possibilities stated above, achieving total gas quality at the weld pool (the point where it counts) can often be an elusive goal. Manifold systems are especially susceptible to contamination because of the typical long length of gas lines from the liquid tank or gas cylinder to the numerous welding stations. Bhadha has addressed these and many other commonly overlooked facts that are influential in creating shield gas contamination [32]. The principal sources of contamination, the gas cylinder and the gas plumbing, are addressed in more detail in the following section.

3.1.1. **Gas Cylinders**

Gas cylinders, when run dry, allow moisture to ingress into the cylinder, causing rust to form on the inner wall. This rust acts like a sponge and soaks up air and moisture during subsequent fillings. It then releases its trapped contaminants into the cylinder gas, which may create a contamination problem that may persist for the entire period of use of the cylinder. Moreover, hydrostatic testing is often employed for cylinders every five years. The testing process, too, may cause problems of rust creation and moisture entrapment.

As moisture can be absorbed readily by most surfaces, if a cylinder valve is left open on a rainy or humid day, the cylinder may become contaminated with moisture. Advances in valve design have recently led to the introduction of minimum pressure non-return valves to completely eliminate this problem.

Suppliers do provide a special brand of cylinders (Aculifed-Specialty Gases) where a special passivating treatment is given to the cylinders to ensure against contamination arising from the cylinder itself. However, these cylinders are part of an elite category that find popularity only in very high purity applications and are ignored by the common fabricator.

In his discussion on "How Shielding Gases are Made", Phelps [33] has addressed the issue of gas purity monitoring procedures and equipment at shielding gas bottling plants. All compressed gas cylinders and manifolded cylinder packages are filled in purpose built computer-managed filling stations. A filling station consists of a series of modules each with the capacity to batch fill fifteen cylinders in a pallet. Empty or partially full cylinders of argon are initially blown down and the waste gas analyzed to determine the condition of the cylinder. If any contamination is found, a cleaning routine comprising several cycles of purging and evacuating with dry clean gas is automatically initiated. Quality assurance systems measure and control impurities such as oxygen and moisture.

Oxygen measurement: To measure oxygen impurities in the ppm range, a sample of the cylinder gas is referenced against a calibrated standard. The gas is passed through a zirconium oxide cell, which generates a voltage proportional to the difference in the oxygen content of the sample to that of the standard.

Moisture measurement: Control of moisture levels in shielding gases is clearly an important issue in order to avoid weld contamination. Moisture can also cause more practical problems such as frozen regulators and internal corrosion of cylinders. "Moisture in the gas" is a common complaint raised by welders

upon having to confront unsatisfactory welding results. In fact, these suspicions could even be substantiated by on-the-spot dew point measurements. The dew point measurement technique relies upon condensation of moisture on a chilled mirror. The lower the temperature of the mirror at which the dew is formed, the drier the gas. Experience, however, has pointed out that it is difficult to measure dew point accurately even with laboratory equipment, and the method is prone to skepticism. One of the main reasons is that the rubber tubing generally employed in dew point devices leads to erratic results due to its high permeability. Moreover, the fact that visual detection constitutes a major step in the measurement adds a factor that varies with the judgement of the operator.

More advanced techniques, however, use dielectric materials which absorb moisture and change capacity to generate a measurable current.

To prevent contamination of inert gas cylinders with other gases, an arrangement is generally agreed upon between the customer and the supplier, whereby the user is requested to leave a certain residual pressure in the cylinder at all times. This assures the supplier that he will not need to purge any contaminating gases from the returned cylinder. The customer is permitted a price adjustment on the basis of the volumes of the gas returned to the supplier. Usually, a residual pressure of 25 psi is sufficient to ensure against contamination of the cylinder prior to recharging.

In summary, although welding difficulty is often attributed to the impurity of the shielding gas, experience has shown that of all possible sources of trouble, the gas in the cylinder itself is least likely to contribute to the difficulty. Hoses and connections that are faulty can readily cause contamination of the

inert shield gas flowing to the torch. The importance of a leak-proof gas delivery system cannot be overemphasized.

3.1.2. Gas Plumbing Material Permeability

Plastic hoses and non-metallic tubing components are commonly used to interface the different parts of a welding system. These components are commonly used between the cylinder and power supply, and between the power supply and the weld torch. Many non-metallic components are quite permeable and allow atmospheric moisture and oxygen to diffuse through their solid walls at the molecular level. Consequently, the gas stream flowing within the lines is contaminated.

Poorly crimped hose connections, loose fittings, or damaged lines serve to aggravate the problem. Bhadha has determined the permeabilities of different hose materials and has made several recommendations regarding the selection of hose material for welding systems [34]. Common materials like PVC or rubber are best replaced by polypropylene or Kel-F 81, materials that have a good resistance to moisture permeation. Table 4 lists the permeability coefficients of different hose materials and can be usefully referred to when selecting the material for a gas carrying system.

Farish [35] has conducted expriments on three hose materials for carrying argon and the results further attest the importance of selecting non permaeable hoses for welding systems. An experiment was conducted with three different hose materials to determine which is most efficient in preventing contamination. The three materials were : polychloroprene rubber,

Table 4 Permeability coefficients of common hose materials

	Common trade name	Permeability Coefficients at 25^{0}C ($P \times 10^{10}$)	
		Oxygen	Moisture
Poly(isoprene)	Natural rubber	23.3	2290
Poly(chloroprene)	Neoprene G	4.0	910
Poly(isobutene-coisoprene)	Butyl rubber	1.3	110
Polyvinyl chloride	PVC (unplasticized)	0.045	275
Poly(tetrafloroethylene)	Teflon	4.2	4.8
Poly(tetrafluroethylene cohexafluropropone)	Teflon (FEP)	4.9	17.0
Poly(ethylene)low density (0.914 gms/cm3)	LDPE	2.2	68
Poly(ethylene)high density (0.964 gms/cm3)	HDPE	0.3	9
Poly(propylene) density 0.907 gms/cm3)	PP	1.2	35
Poly(vinyledene chloride)	Saran	0.005	0.5
Poly(trifluro chloroethylene)	Kel-F 81	0.04	0.1
Poly(ethyl metahcrylate)	Plexiglass	1.2	32000
Poly(carbonate)	Lexan	1.4	1400
Poly(ethylene terephthalate)	PET	0.035	130

Note: Permeability coefficient = $\frac{\text{(Amount of permeant) (Film thickness)}}{\text{(Area) (Time) (Pressure-drop across film)}}$

Units of P: [cm^3 cm] / [cm^2sec (cm Hg)]

Source: Bhadha, P.H., "Effect of Weld Torch Hose Materials on Shielding Gas Quality", paper to be published

polytetrafluroethylene, and polyolefin. Argon was passed through 147 feet long hoses for 100 h at rates of 0.9 and 4.7 cfh. (These flow rates were considered representative of purging and shielding conditions, respectively.) Moisture readings were taken at both ends of the hose. The argon flow initially dries the mositure accumulated in the hose, but then the permeation of moisture through the hose material proceeds at a rate that prevents further drying. The polychloroprene material performed the worst under these condidtions, while the polyolefin material performed the best.

3.1.3. **Leaks In Gas Lines**

It is commonly believed that a given gas line under pressure will automatically safeguard against the diffusion of atmospheric moisture. However, examination of table 5, from the work of Bhadha [32], indicates that there does exist a tremendous driving force for the back diffusion of atmospheric moisture into the line carrying the process gas. It is erroneous to assume that just because the sample line is under pressure, no atmospheric moisture can diffuse into the lines.

The following calculation, by Bhadha, although a gross simplification, does impress the fact stated above.

"Consider a case where the process gas has a moisture content of 10 ppm (not uncommon in regular welding grade gases) equivalent to a dewpoint of -60^{0}C and the tempearture of the surrounding air is 25^{0}C with a relative humidity of 50%. Under these conditions, the moisture content of the atmosphere would be 16,500 ppm (by volume)! Consequently, there is a tremendous driving force for the back diffusion of atmospheric moisture into the process gas.

Table 5. Driving force for atmospheric permeation into gas delivery hoses

Species	Conc. of species inside hose (ppm)	Conc. of species outside hose (ppm)	Driving force for species to diffuse out of hose to atm. (ppm)	Result
Argon	950,000	9000	~ 940,000	Argon diffuses out of hose
Helium	50,000	5	~ 50,000	Helium diffuses out of hose
Nitrogen	80	~ 780,000	~ 780,000	Nitrogen diffuses in to hose
Oxygen	20	~ 209,000	~ 210,000	Oxygen diffuses in to hose
Moisture	10	~ 20,000	~ 20,000	Moisture diffuses in to hose

Note: Large driving force for permeation of oxygen, moisture, and nitogen from the atmosphere in to the hose

Source: Bhadha, P.H. "Effect of Weld Torch Hose Materials on Shielding Gas Quality", paper to be published

A simplified equation to estimate the leak across a concentration gradient is given by the follwing equation:

$$C/C_0 = e^{-VX/D}$$

where C and C_0 are the concentrations across a barrier
(= 10 ppm and 16,500 ppm for the above case)
X is the length of the leak (assumed to be equal to the wall thickness of the tube , 0.035")
D is the diffusivity of moisture in air = 0.25 cm^2/sec at 25^0C
V is the streaming velocity, cm/sec
For a leak across a 1/4" tubing with a 0.035" wall thickness, the streaming velocity, calculated by the above equation, would be approximately 21cm/sec. Assume the source of the leak in this case was a hole 0.001 inch in diameter in the tubing. For this case, the leak cross section area would be 0.000005 cm^2 and the moisture leakage would be 0.00011 cm^2/sec (obtained by multiplying the streaming velocity by the leak cross-sectional area).

Assume the process gas is flowing at a mere 2 cfh, the mositure contributed by the leak would be 7 ppm.!"

This sample calculation demonstrates the significance of seemingly insignificant leaks.

3.2 **Shielding Gas Contamination Effects on Important Alloys**

The issue of welding shield gas contamination by a different variety of impurities has been addressed only superficially by the academic and welding process community. The efforts undertaken so far have only concentrated on the understanding of the effects of gross atmospheric contamination of shield gas in the welding of common metals such as aluminum and steel. A brief review of the effect of shielding gas and its purity on the weld behavior of various alloys is presented in the following section.

3.2.1. **Aluminum**

Devletian and Wood [36] have conducted a literature review to determine the factors affecting porosity in welds deposited by the GTAW and GMAW processes. Hydrogen was established to be the dominant cause of porosity in aluminum alloy welds. Aluminum alloys are far more susceptible to the formation of porosity than all other structural materials. Susceptibility to pore formation stems from the fact that even trace levels of hydrogen exceed the threshold concentration needed to nucleate bubbles in the molten weld pool. Factors such as shielding gas mixture and external pressure influence the size, distribution, and the amount of hydrogen pores generated in the weld.

Within the welding community, Torres et al. [37] have quantified the effects of shielding gas contamination in variable polarity plasma arc welded aluminum. (The VPPA process has completely replaced GTAW in many of NASA's space shuttle fabrication applications.) The effects of contamination were analyzed mainly in terms of micro porosity, incomplete fusion, undercutting, and surface rippling. Impurities in either the arc or the shield gas are expected to be particularly important in plasma arc welding for the following reasons:

1. The high weld speeds attainable provide less time for impurities absorbed from the gas to effervesce.
2. The large contact area between the plasma gas and the work piece during 'keyholing' permits easy absorption of impurities from the gas into the weld pool.

3. The smaller weld puddle suggests that super saturation of gases such as hydrogen can take place more easily and cause porosity on cooling.
4. Since an entire keyhole must re-fuse after the torch passes, surface active gases may alter flow patterns and cause undercutting or other weld defects.

In the experiments performed, defects such as porosity, asymmetric undercutting, incomplete fusion, and fine extrusions at the edge of the weld were associated with various levels of hydrogen, methane, nitrogen, and oxygen contamination in the shielding gas admixture. The defects produced by various contaminant levels are illustrated in figure 3.

In general, when welding aluminum, the hydrogen formed during the dissociation of moisture in the gas and the air surrounding the arc are considered to be the main cause of weld pore formation. However, a second cause is the moisture introduced in the molten metal by the film of oxides on the base metal and the electrode wire. Psaras [38] has examined the effects of moisture content and dry hydrogen added to argon shielding gas on the formation of pores in aluminum welds. The research proved that although the addition of water vapor to the argon promotes pores to rapidly form in the weld, the addition of dried hydrogen to the argon does not increase the volume of cavities in the weld metal. For example, when metal is deposited with a consumable electrode in moistened argon with dew points between -15 and -30^{0}C, depending on the external component of the arc length, the total volume of cavities in the weld metal equals 5-9 cc/100g of weld metal. The larger the surface of contact between the arc and the moistened shielding gas, the larger the number of pores formed in the weld metal.

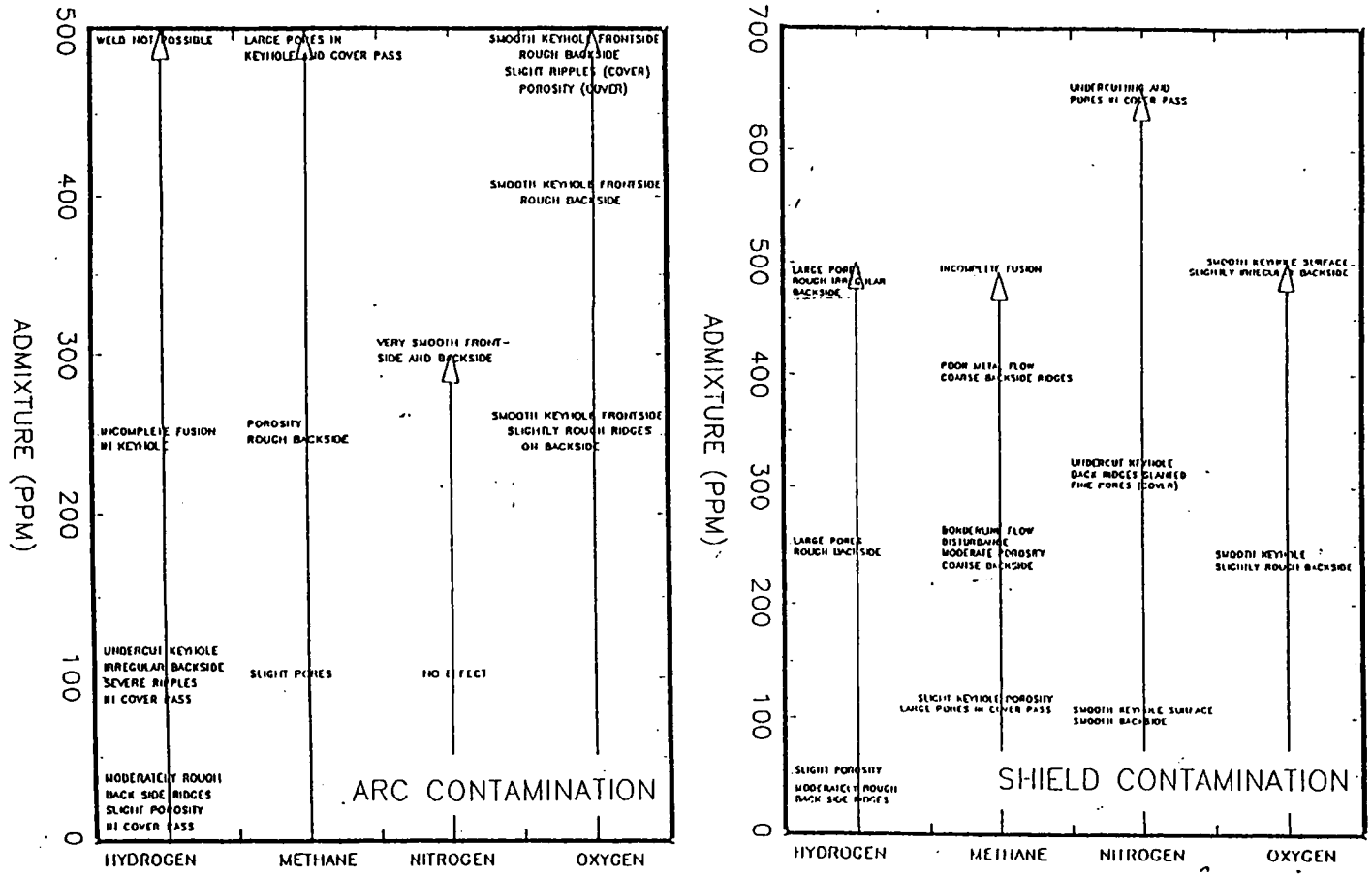


Figure 3. Effect of gas contamination in VPPA welded aluminum

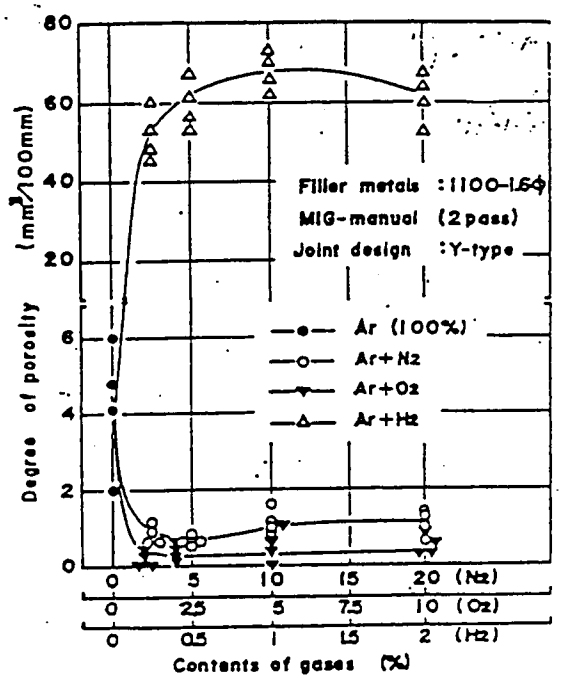
Source: "Gas Contamination Effects in VPPA Welded Aluminum", Torres et al, Welding Journal, November 1990, pp 43-45

Fukui, Sugiyama and Terai [39] studied the effects of nitrogen, oxygen, and hydrogen gases added to argon gas on the weldability and properties of MIG welds in commercially pure aluminum and its alloys. They concluded that the addition of up to 10% nitrogen was effective in practical applications in increasing the depth of penetration and tensile strength, and refining the structure in the welds. In commercially pure aluminum, nitrogen decreased the porosities in the weld with little formation of scale on the bead surface; in Al-Mg alloys, nitrogen formed brown scale on the bead surface and the porosities were not always decreased.

The addition of up to 5% oxygen was considered to be advantageous in practical applications because it increased the depth of penetration and decreased the porosities in the welds. However, its addition over 5% was harmful because of the occurrence of puckering and the formation of excessive oxide film on the bead surface.

The addition of hydrogen was harmful because it remarkably increased the porosities, resulting in the loss of weld mechanical properties.
The effect of gas composition on the porosity ratings of 1100 and 5083 aluminum alloy welds is displayed in figure 4. (Incidentally, both materials were also included in the current work.)

Bicknell [18] has experimented with the use of argon/freon shielding gas mixtures on the GMA welding of aluminum. Similarly, Koyangi [40] has studied the effect of adding chlorine to shielding gas in the GTA welding of aluminum.



Welding Procedures		Pass	1100				5083			
			Ar	Ar+N2	Ar+O2	Ar+H2	Ar	Ar+N2	Ar+O2	Ar+H2
Bead-on-plate (machine)		1	◑	◔	◔	●	◑	◕	◔	●
Butt-joints	MIG-machine	2	◑	◔	○	●	◑	◕	◔	●
	MIG-manual	2	◑	◔	◔	●	◑	◔	○	●
	MIG-manual	3	◑	◑	○	●	◑	◑	○	●
	MIG-manual	4	◑	◔	◔	●	◑	◔	◔	●

○ Very good ◔ Good ◑ Fair ◕ Poor ● Very poor

Figure 4. Effect of gas composition on the porosity rating of 1100 and 5083 aluminum welds

Source: "Effects of Nitrogen, Oxygen, and Hydrogen Gases Added to Argon Gas on MIG Welding of Aluminum", Fukui, et al., Transactions of Japan Welding Society, Vol. 1, No.1, April 1970

3.2.2. **Duplex Stainless Steel**

Geipl [17] has investigated the application of GMAW welding for welding corrosion-resistant duplex steels. Argon gas mixtures with low carbon dioxide additions are considered beneficial in terms of their application. Inert gases result in poor arc stability and their use is restricted to few applications. Ternary Ar-$C0_2$-He mixtures help improve the wetting action.

Strom and Li [41] have studied oxide-induced corrosion in GTA autogenously welded SS 2352 and SS 2353 stainless steel. The shielding gas used on the root contained 2, 26 and 99 ppm oxygen. Examination of the pitting corrosion potential enabled the following conclusion to be drawn: Welding with higher oxygen content in the shielding gas implies lower pitting corrosion resistance. CPT values for welds that had been shielded with 2, 26, and 99 ppm oxygen were 17.5, 15, and 5-10^{0}C, respectively. The results indicate that welding with shielding gas containing a maximum of 30 ppm oxygen does not substantially affect the pitting corrosion properties.

Stekly et al. [42] have studied the relationships between various welding techniques (MMA, MIG, FCAW) on the properties of super duplex stainless steels with special emphasis on the recovery and significance of nitrogen, oxygen, and carbon concentration. It was found that in flux cored arc welding, the recovery of these elements is virtually insensitive to welding conditions and the shielding gas composition.

Bonnefois [43] has proved the beneficial effects of nitrogen additions on the microstructure stability and on the pitting corrosion of S31803 and S32550 duplex welds. Mudali et al. [44] have conducted similar work on austenitic

steels to assess the effect of introducing nitrogen into the weld pool with the shielding gas. With increasing nitrogen levels, the delta ferrite contents in samples of 316 and 304 SS weld metal decreased from 3.8 to 0 FN.

3.2.3. **Titanium**

Titanium is being increasingly used in the chemical and power industries for its outstanding corrosion resistance in certain chemical and marine environments. It has been widely studied by many welding researchers. Morgan [45] has studied the application of gas analysis to the monitoring and control of shielding atmosphere during welding, which enables the atmosphere purity to be specified. Titanium, although readily weldable, reacts strongly with oxygen and nitrogen at elevated temperatures, leading to severe hardening and embrittlement. The dependence of weld mechanical properties on shielding gas purity is illustrated in figure 5.

In 1954, Barret and Lane investigated the effect of atmospheric contaminants on arc welds in titanium [46]. Helium-based shielding gases included in the study contained 0.25% to 1% oxygen, nitrogen, air, and 0.63 to 2.53 mg of water per liter. Weld properties were evaluated by tension, bend, hardness, and impact tests. Among other findings, it was concluded that nitrogen, oxygen, and dry air adversely affected the properties when their presence was as low as 0.25% in the weld atmosphere. Heavier gages were more sensitive to oxygen and nitrogen since the last passes covered a large surface area. Impact strengths were considerably lowered by small volume percentages of these contaminants. Surprisingly, the study also reported that

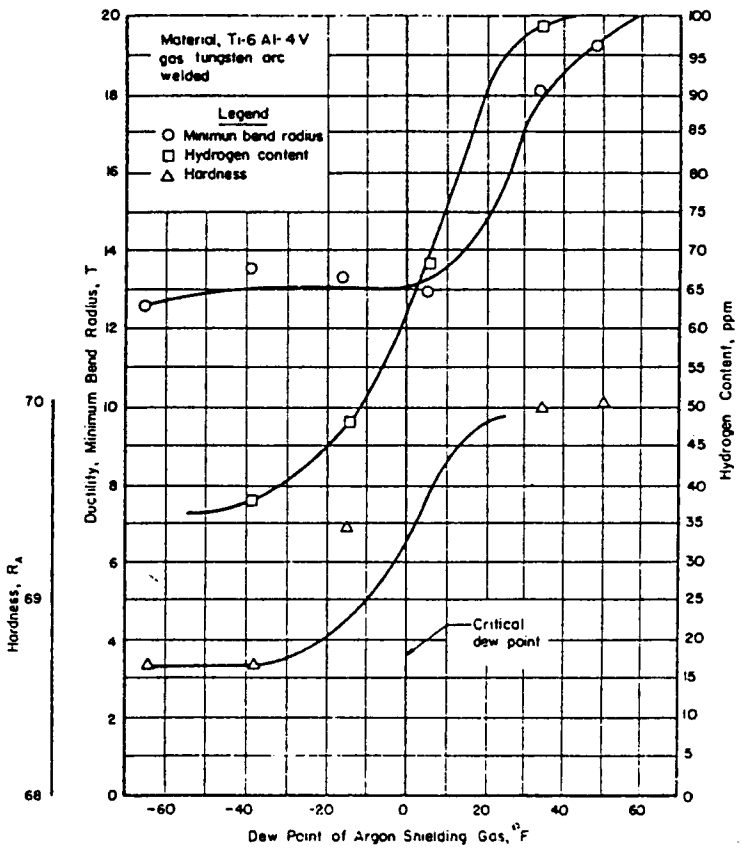


Figure 5. Influence of shielding gas purity on the mechanical properties of titanium welds

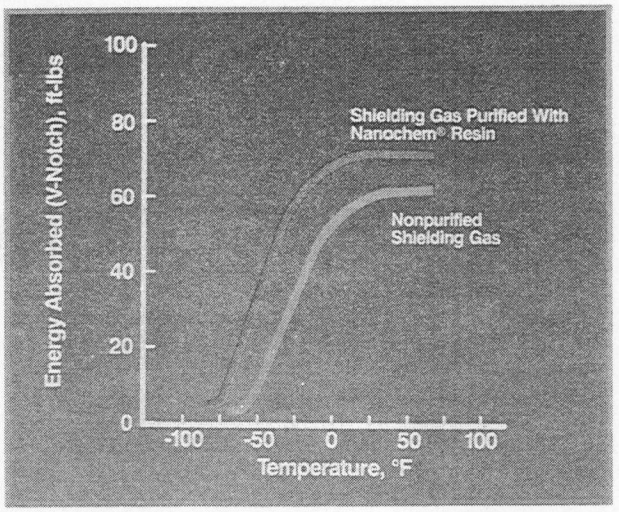
Source: Welding Handbook, Metals and their Weldability, 6th edition, section 4

hydrogen and moisture in the welding atmosphere had little effect on the weld properties, including impact strength.

3.2.4. **Ferralium 255 and E-Brite**

The studies of Krysiak and Bhadha on Ferralium 255 and E-Brite are, in fact, the only work executed so far to exclusively tackle the issue of the effect of shielding gas purification on weld behavior [47]. The work was the outcome of tests conducted to evaluate the effect of using the Hercules-developed Nanochem gas purification system. Ferralium 255 and E-Brite were welded using the gas tungsten arc welding process using different levels of shield gas impurities (40-42 ppm moisture and 18-20 ppm oxygen). The employment of the Nanochem gas purification system was effective in removing the moisture and oxygen to less than 10 ppb (limit of sensitivity of the equipment). Notable observations made were the clarity of the weld pool, which was totally free of floating particulates (oxides), and a marked improvement in wetting. The improved wetting suggests a reduction in the viscosity and/or surface energy due to impurity removal from the weld pool. Effectiveness of purification was also quantified in terms of improved lateral expansion and percent ductile shear fracture. The charpy impact results of GTA welded Ferralium 255 and E-Brite 26-1 welds are displayed in figures 6a and 6b, which substantiate the potential for weld properties improvement by shield gas purification. It was from this point that the UTK-Hercules project was initiated and the program has further quantified the effects of shield gas purity as applied to the welding of other popular industrial alloys.

(a)



(b)

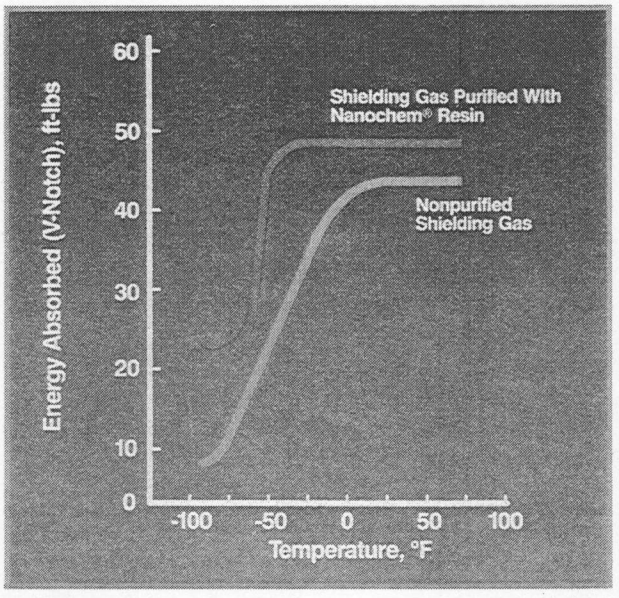


Figure 6. Hercules test results indicating improvement in charpy toughness of GTA welds shielded with Nanochem purified gas. (a) Ferralium 255 (b) E-Brite.

Source: "Shielding Gas Purity Improves Weld Quality", K.F Krysiak and P. M. Bhadha, Welding Journal, November 1990

3.2.5. **Steel**

Of all elements found in air, oxygen, nitrogen, and hydrogen cause the most difficulty in the welding of ferrous materials. Oxygen, by virtue of its higher reactivity, combines with other elements in the metal to form undesirable oxides and gases. Nitrogen, too, can cause a serious problem in the welding of steel. Owing to its high solubility in iron at elevated temperatures, it precipitates out of iron as it cools, forming detrimental metal nitrites. These nitrites cause high yield and tensile strength, and increased hardness. This condition, in turn, lowers ductility and impact resistance and often leads to weld cracks in and near the weld zone. Hydrogen reacts in a similar fashion by dissolving in the molten metal at elevated temperature and escaping as the metal solidifies. As the rejection of gas continues, little bubbles of hydrogen are trapped in the frozen metal, creating high pressures and stresses. These stresses subsequently lead to micro cracks, larger cracks, and underbead cracks. In addition to causing cracking, porosity, and oxidation, gas contamination is also responsible for arc instability and degradation of mechanical and corrosion properties.

Tuthill [48] has addressed the effects of moisture in the CO_2 welding of steel. A laboratory setup allowed the addition of moisture to dry carbon dioxide gas to produce shielding gas with dew points varying from - 43^0 to + 90^0F. For weld metal specimens welded with this wide range of moisture contained in the shielding gas, testing indicated that high moisture did not affect the yield strength, hardness, or ultimate strength. It did, however, affect the percent elongation. Spatter did increase with the addition of moisture to the shielding

gas. It appeared from the tests that an effort should be made to keep the dew point at -20^{0}F or below. It was also observed that in the commercial grades of CO_2 selected for the study, not all cylinders met the above stated requirement, although they did not appear very far from the desired value.

Krahl [49] has quantified the influence of humidity on welding behavior, hydrogen content, density, and oxidation of deposited steel. He employed MIG/MAG welding processes using the protective gases argon and carbon dioxide humidified to a defined extent under atmospheric pressure in accordance with saturation temperatures of 10, 20, and 30^{0}C. Oscillograms for the arc voltage and welding current indicated that the presence of steam in the protective gas affected the progress of the welding process. The arc became increasingly unstable with an increase in the number and extent of voltage and current dips per unit time. The hydrogen charge in the weld deposit also increased linearly with an increase in atmospheric humidity. Comparison of the silicon and manganese contents in the wire electrode and in the various specimens produced in different arc atmospheres indicated that when welding under a humid protective gas, a melting loss of alloying elements subject to intensive oxidation can be expected.

3.2.6. **HY Steels**

Nippes [50] has studied the critical hydrogen content required to initiate cracking in welds of HY-130 steel. For AX-140 welds on HY-130 steels, the critical hydrogen content was determined to be 1 ppm. Matsuda [51] has studied the effect of oxygen content on weld metal diffusible hydrogen in GMA

welded HY-130. If the shielding gas contains hydrogen or moisture, an increase in shielding gas oxygen content or weld metal oxygen content increases the diffusible hydrogen content. Lowering the partial pressure of water vapor in the shielding gas decreases the effect of oxygen. For GMA welding HY-130 steel, the dew point of the argon-oxygen shielding gas should be kept as low as possible (-70^0C).

North et al. [52] are currently studying weld metal cracking in HY-100 and HSLA-100 type steels and are examining the role of oxygen in promoting hydrogen cracking susceptibility using laboratory cracking tests (the LB-TRC test method). Oxygen is found to promote hydrogen cracking at prior austenite grain boundaries and detailed microscopy is being used to analyze the effect.

3.2.7. **Inconel 600**

Savage and Lundin [53] have investigated the GTA process for welding Inconel alloy 600. In their studies, two different gas cylinders, both labeled 99.99% argon, resulted in significant differences in weld penetration. Mass spectrometer analysis revealed that even a small amount of impurity elements in the shield gas, especially oxygen, had a strong effect on the melting efficiency of the GTA process. An increase in the total impurity content in argon from 690 to 2440 vpm caused a 50% decrease in penetration and also resulted in a considerable portion of the puddle surface being covered with a blanket of oxide.

3.2.8. **High Alloys**

Geipl [54] has studied the orbital GTA welding of electropolished high alloy steel tubes. Criteria investigated were type of shielding, purging, backing gases, purge flow rate, and velocity of flow. The efficiency obtained in the purging of pipes was not proportional to the purge gas flow rates. The relationship between flow and time was not linear. When acceptable oxygen contents (50-100 ppm) were obtained inside the pipe, no visible oxidation was found in the weld area. Varying amounts of moisture and oxygen pickup were found in plastic hose lines of varying quality. Levels were reached that no longer met the requirements for shielding and backing/purging gases. These high alloy steel tubes are used in the pharmaceutical and food industries and especially in the supply of high-purity gas for microelectronics. Therefore, it is especially important to avoid formation and accumulation of contaminants and deposits when welding. Geipl has also discussed the deleterious effects of the weld and root shielding gases, gas rinsing, and oxygen absorption due to inadequate weld preparation.

3.2.9. **316 Stainless Steel**

Kujanpa [55] examined the role of shielding gases in discontinuity formation in GTA welding of 316 austenitic stainless steel strips. The solidification cracking observed was entirely due to shrinkage upon weld cooling and hence no relation was found between cracking and shielding gas. However, center cavities and undercut depths were found to be affected by the

type of shielding gas used. Since undercut depths increase with the welding current, an arc shielding gas that promotes deep penetration allows the use of lower welding currents and hence results in shallower undercuts. Similarly, the use of an Ar-H_2 or Ar-He gas reduces the volume of the weld pool needed for complete root penetration, causing a lower amount of center cavities. The study did not address the aspect of impurity contents in the shielding gas and their possible effects.

3.2.10. **Molybdenum**

Lim and Kewon [56] studied the appropriate welding conditions for a molybdenum heating element used in a hydrogen annealing line. It was found that when the shielding gas contained 0.3% oxygen, center bead cracking appeared during weld metal solidification. Argon of high purity was recommended (99.999%) to alleviate the problem.

3.3. **Moisture Purification Systems**

Several moisture purification devices exist that may be used for inert shield gas purification [32]. The advantages and drawbacks of some of these systems are elaborated upon below.

3.3.1. **Molecular Sieves**

Molecular sieves belong to a class of compounds known as zeolites. In zeolites there is a framework of silicon-oxygen and aluminum-oxygen atoms that form a structure honeycombed with relatively large cavities. By selectively adding or removing other metal atoms such as sodium, potassium, or calcium, the size of these cavities can be tailored to allow molecules of only a specific size to pass through. A pore size of 4 angstroms gives 4A molecular sieves, which are often used for the removal of moisture from inert gases.

Molecular sieves need to be heated to a temperature of 30-400^{0}C by passing an extremely dry gas through them before they can be used. They can also be regenerated by the same procedure, which is an advantage provided that the user has the necessary expertise and the ability to produce the dry gas. There are two key disadvantages with molecular sieves. One, the outlet moisture concentration gradually increases as the molecular sieve-based purifier is being depleted. Two, unless the exit gas from the purifier is continually monitored there is no indication when the molecular sieve-based purifier is spent.

3.3.2. **Dessicants**

The two desiccants, silica gel and calcium sulphate treated with cobalt chloride find use in a number of applications. Silica gel is widely used for packaging materials for shipment, but is unsuitable for drying gases to the low

part per million levels necessary for welding processes. There is also no indication when the material is spent.

Cobalt chloride treated with calcium sulphate (Drierite) is used by some researchers because it undergoes a color change from blue to pink in the presence of moisture. Unfortunately, this color change occurs only at very high moisture concentrations (300-800 ppm). In comparison, standard welding grade argon is 99.9995% pure and is certified to have a maximum impurity content of 50 ppm. The typical moisture concentration in the gas is about 5-20 ppm. As a result, the welder cannot rely upon such dessicants to either dry his shielding gas or to indicate when the purifier needs to be replenished.

3.3.3. **Getters**

A hot titanium sponge has been used for purification of gas to dry boxes. The sponge is maintained at a very high temperature of about 80^0-1000^0C. The key advantage to using such a getter is that trace levels of nitrogen (besides moisture, oxygen, and other impurities) are also removed. Other "non-evaporable" getters based on zirconium oxide and vanadium oxide are also available that operate at somewhat lower temperatures, 350-600°C. Another advantage of such getters is that they can be regenerated, but with some loss of capacity (total quantity of impurities removed before the purifier is spent). Unfortunately, very large and expensive getter-based purifiers are necessary to handle the relatively large flow rates 25-80 cfh typically used for efficient shielding. There is also no fool-proof method to indicate when the purifier is spent.

3.3.4. **Nanochem Purification System**

The Nanochem purification system used in this program is a resin-based purification system. This class of resin-based purifiers depend on chemical resins for filteration of impurities. The working of the Nanochem purification system used in this program is addressed in detail in the following section.

3.3.4.1. **Principles of Operation**

The Nanochem gas purifier consists of active proprietary alkali compounds on a polymeric substrate of a large surface area [57]. An external view of the Hercules, Inc. developed product is presented in figure 7. There are several advantages offered by this resin-based system:

1. Room temperature operation.
2. Self indication: The resin undergoes a color change from black to brown when spent.
3. Capability to remove a large variety of impurities such as oxygen, moisture, carbon dioxide, and hydrocarbons to very low levels-up to 1-10 parts per billion. It is important, however, to recognize that Nanochem purification is not recommended for weld shielding gases that utilize additives such as oxygen and carbon dioxide. This restriction stems from the fact that the additives are filtered out, and only the base inert gas (argon/helium) is let through. Moreover, care should be taken in



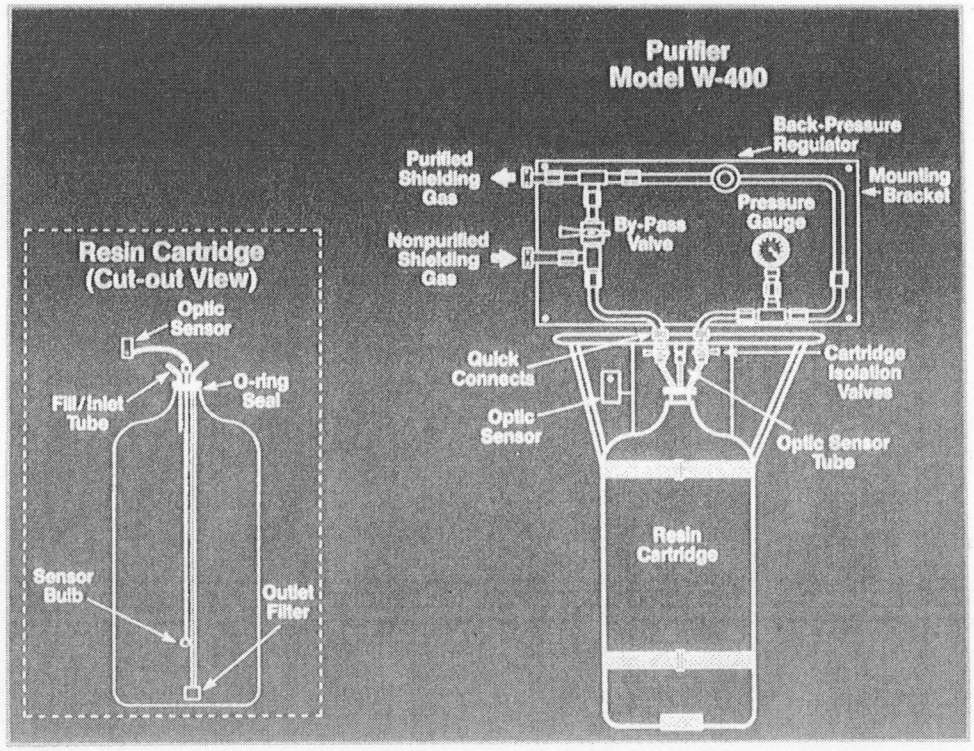
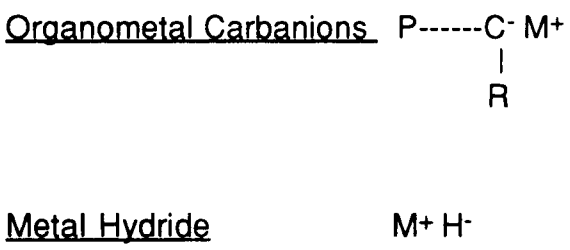


Figure 7. The Nanochem resin-based, gas purification system

the case of non-compatible gases, such as carbon dioxide, that can react with the Nanochem resin and result in a highly exothermic reaction.

4. The system offers consistent purification irrespective of high impurity surges that may result from leaks or even an improper cylinder change operation.

There are two types of functional groups in Nanochem:



(H = Hydrogen, C = Carbon, P = Polymer, R = H, CH_2, C_2H_6)

The carbon in the carbanion and the hydrogen in the hydride carry a net negative charge. Negatively charged carbon and hydrogen ions are unstable, and, as a result, react with other elements to return to their normal oxidation states. The subsequent reaction is the principle by which the Nanochem resin reacts with other impurities.

The resin can react with a number of different impurities through various reaction pathways. They are:

1. Reduction
2. Deprotonation
3. Nucleophilic addition and substitution:

1. Reduction

Oxygen gets reduced (its valence state goes from 0 to -2). The by-products are oxides and hydroxides:

$$P\text{----}M + O_2 \text{-----> } 2MOR$$

$$2MH + O_2 \text{-----> } 2MOH$$

2. Deprotonation

A proton (H^+) is removed from H_2O (which can be expressed as H^+OH^-). The by-products are metal hydroxide and hydrogen:

$$P\text{----}RM + H_2O \text{-----> } P\text{----}MH + MOH$$

$$2MH + H_2O \text{-------> } 2MOH + H_2$$

3. Nucleophilic Addition and Substitution

Carbon dioxide and carbon monoxide react with the hydride groups to form carbonates and bicarbonates:

$$MH + CO_2 \text{-----> } M_XCO_3, M_YHCO_3$$

$$MH + CO_2 \text{-----> } M_XCO_3, M_YHCO_3$$

$$[X, Y = 1, 2]$$

Thus, gaseous impurities--oxygen, water, carbon dioxide and carbon monoxide-- are converted into nonvolatile solids, such as oxides, hydroxides and carbonates that stay chemically bonded to the resin.

Given the number of reaction pathways by which the resin can react with impurities, it is not surprising that the resin can react with a large multitude of impurities. The reactions are so irreversible that there is essentially no impurity

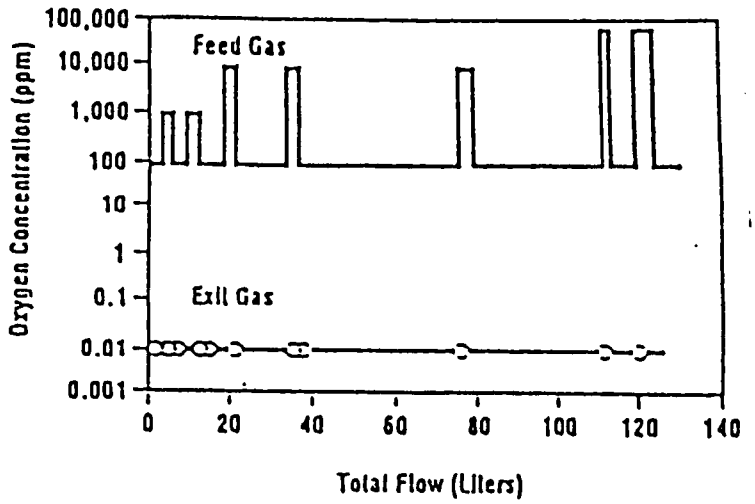
left in the purified gas. Figure 8a shows the removal of oxygen from nitrogen by Nanochem resin. Oxygen surges as high as 50,000 ppm are completely removed to levels less than 10 ppb. Figure 8b shows the purification of nitrogen containing 7 ppm moisture by a Nanochem resin-based purifier. Once the gas is sent through the purifier, the moisture level decreases rapidly to about 1 ppm. The slow decrease in the outlet moisture concentration below 1 ppm is due to the slow drying of the gas sampling lines to the moisture meter and the gas delivery lines inside the moisture meter.

Two models of the Nanochem gas purification system are available, which differ primarily in the use level for which they are designed. Model W-400 is intended for the user whose arc time is 1,000-2,000 hours per year at typical flow rates of 15-30 cfh; and the W-400-A, for arc times greater than 5,000 hours at 30-60 cfh. Both units are installed in the shielding gas line between the welding power supply and the torch hose.

3.3.4.2. **Nanochem Applications**

The Nanochem resin-based purification system finds application in semiconductor industries, welding industries, gas chromatography areas, particle accelerators, and in laboratory applications using nitrogen and argon. Some of the applications in the welding area where Nanochem has been successfully applied are the welding of space shuttle tanks, aircraft components, jet engines, turbine repair, fiberoptic cables for ocean communications, cables for microwave communications, process equipment, industrial knife blades, and golf clubs. Some applications where Nanochem

(a)



(b)

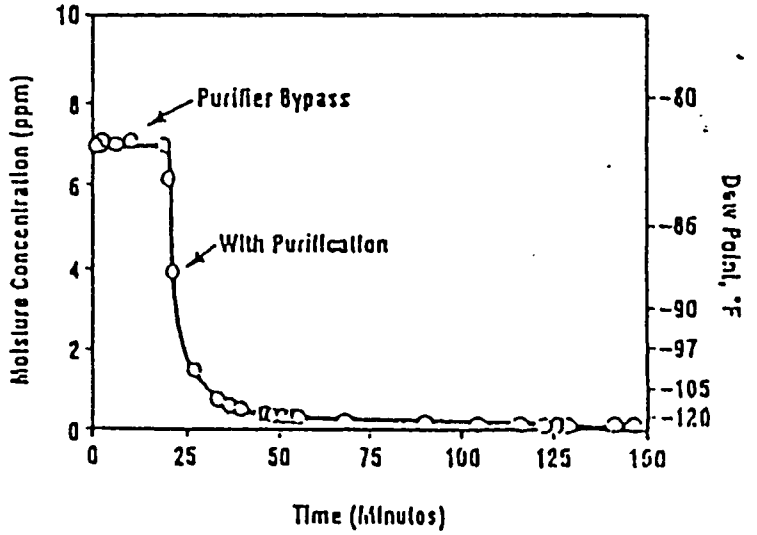


Figure 8. Removal of impurities from nitrogen using Nanochem.
(a) Oxygen (b) Moisture

Source: "Purification to PPB and PPM Levels", P. M. Bhadha and C. L. Cowan, 1994 Proceedings-Institute of Environmental Sciences

purification is beneficial are found in the semiconductor industry [58, 59, 60], where autogenous orbital gas tungsten arc welds connect tubes and fittings that deliver gases for processes which have no tolerance for contamination arising from the weld area.

3.4. **Gas-Injection**

In an effort to enhance weld properties and arc characteristics using super-pure gas, this study also examined weld gas delivery systems for possible modifications and improvements.

Efforts taken in this direction were based on a patent filed by Copher, Krysiak and Paoletti in 1990 [61]. The patent utilizes the concept of passing a stream of shielding gas into the liner/sleeve that carries the filler wire in a gas-shielded arc welding torch such that the gas exits both ends of the sleeve-the contact tip end and the wire inlet end. (Figure 9). This configuration keeps atmospheric air out of the sleeve and the positive pressure created at both ends of the sleeve (wire entry and exit points) prevents harmful contaminants from reaching the weld pool through the sleeve and causing sputtering and imperfect welds. The same or different shielding gases can be passed inside and outside the sleeve in accordance with the invention. When this modification (hereafter referred to as gas-injection) is used in concert with gas purification systems such as the Nanochem resin-based system, the weld pool is theoretically free of any harmful contaminants.

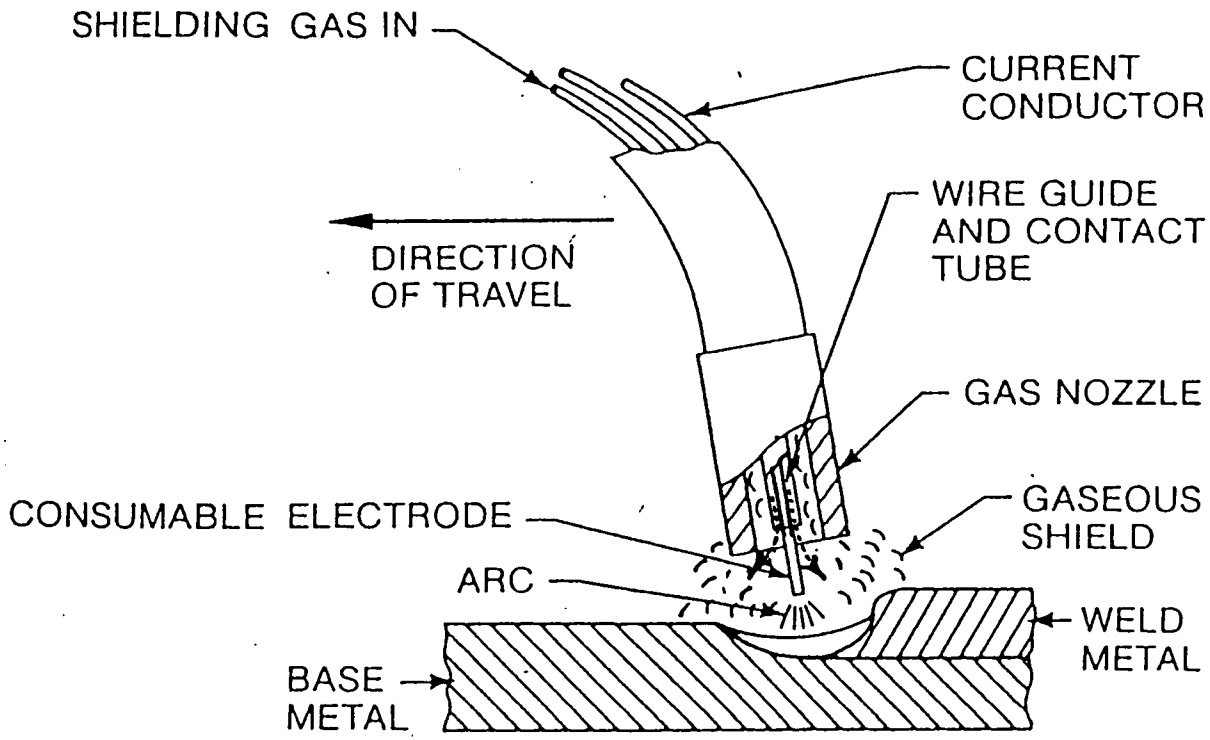


Figure 9. Gas-injection configuration showing pressurization of the wire guide tube with inert gas to prevent air-entrainment and weld pool contamination (dotted lines)

CHAPTER 4

EXPERIMENTAL SETUP

4.1. Welding Equipment

New state-of-the-art welding equipment was purchased and set up for conducting the experiments required for the study. The capabilities of the equipment are as follows:

4.1.1. Gas Metal Arc Welding

Wire feeder: Miller S-64M

Microprocessor-controlled constant speed wire feeder

GMAW, GMAW-P and FCAW capabilities

0.6 to 3.2 mm (0.023 to 1/8 inch) wires

Standard wire feed speed of 50 to 780 ipm (1.3 to 19.8 mpm)

Programmable pulse weld parameters

Power source Miller Maxtron 450

CC/DC and CV/DC power source

Multi-process capabilities

450 amperes at 100% duty cycle

Miller XR Push-pull feeder system: (Aluminum welding)

115 Volts AC wire feeder and 30 volts DC gun

Compatible with Maxtron 450 wire feeder

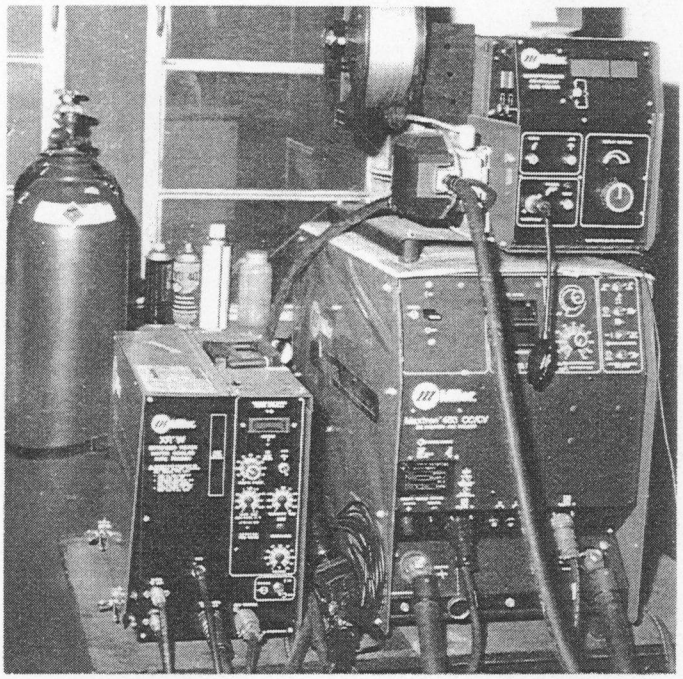
70 to 875 ipm (1.9 to 22.2 mpm) wire feed speed range

0.030-1/16 in aluminum and .030-.045 in hard or cored wire

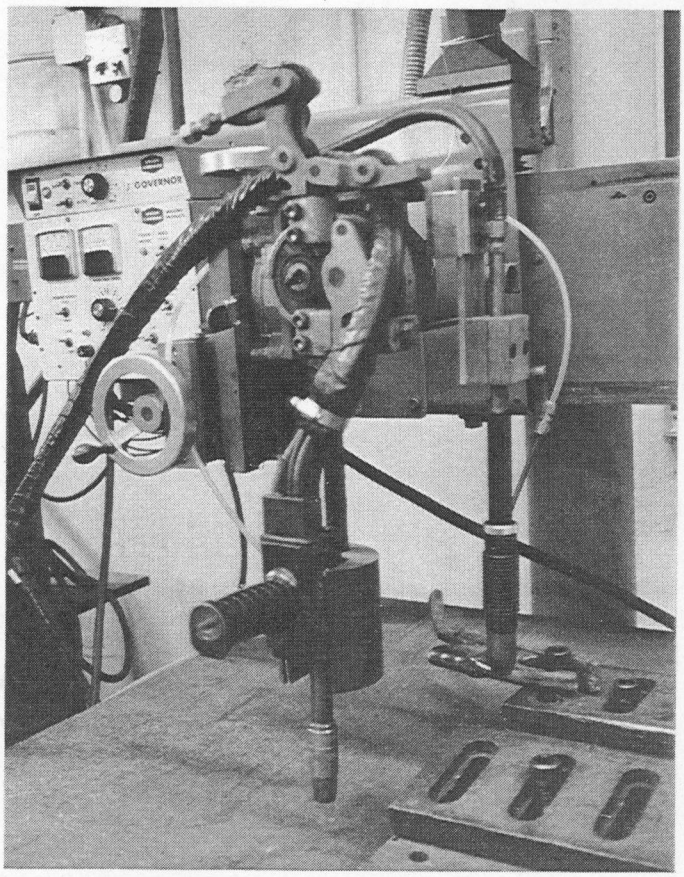
Water-cooled push-pull gun

4.1.2. **Gas Tungsten Arc Welder**

The fully automated welding system consists of a power supply, a fully programmable controls console, and a track mounted welding head. The welding head is equipped with a carriage drive, wire feed, cross seam oscillator, dwell capability, and an automatic voltage control. Photographs of the welding equipment used for this investigation are displayed in figures 10 through 12. Given the vast multitude of variables that affect weld quality, it was essential that only automatic welding be employed in a study of this nature. This eliminates discrepancies arising from operator variations. All torches were mounted on automatic carriage systems, and the GTA and GMA welding processes were both automated. The adaptation of the Nanochem purification unit and gas blending facility to the GTA and GMA process equipment is illustrated in the flow diagrams shown in figure 13 (a, b, & c). The gas supply systems were modified such that the gas could be fed directly to the torch, independent of the welding machine. This modification provided more stability for gas blending, allowed extended purging times and more flexibility in pressure and flow regulation.



(a)



(b)

Figure 10. Gas metal arc welding equipment.
(a) Power source and wire feeders (b) Weld torches

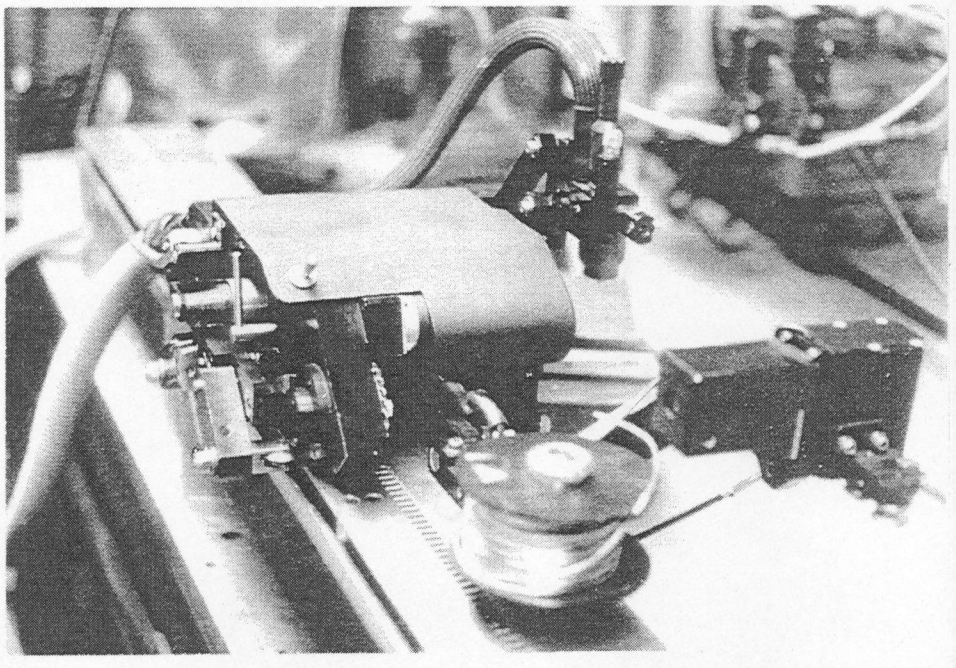


Figure 11. Automatic weld-torch head of gas tungsten arc weld system

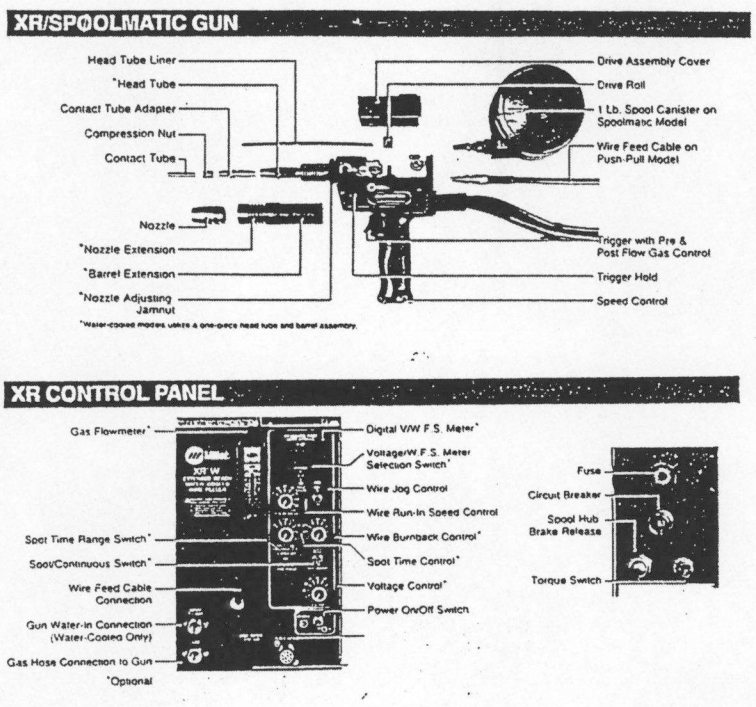
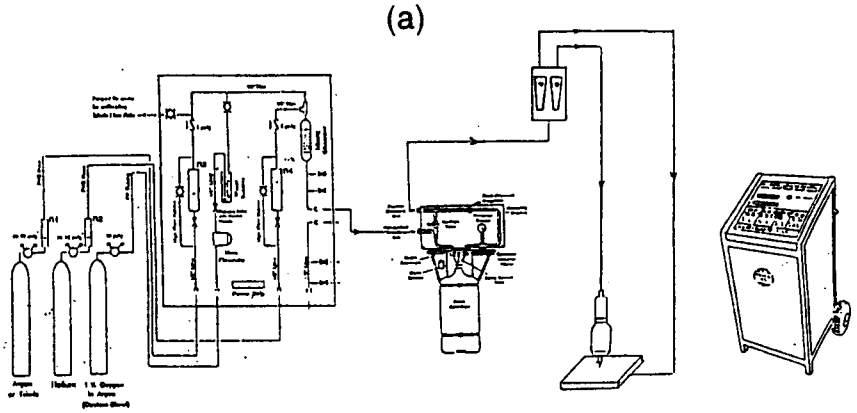
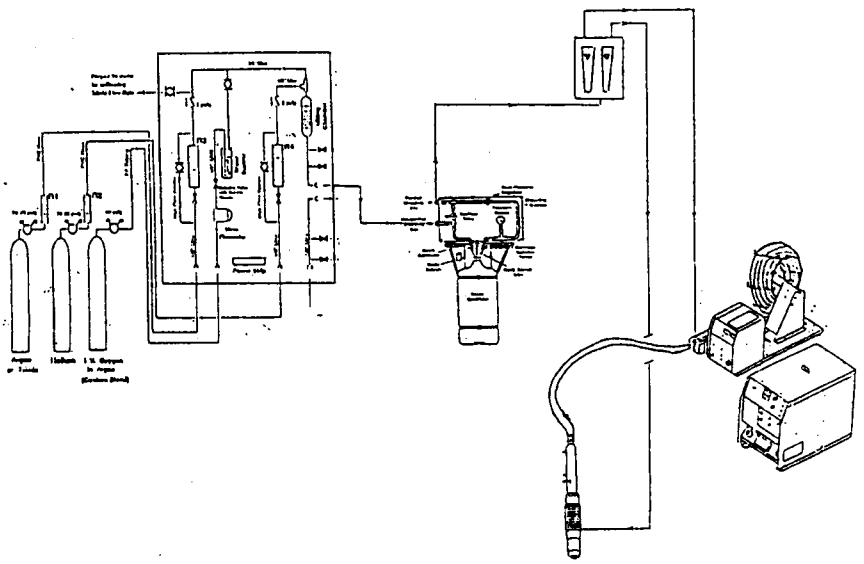


Figure 12. Gas metal arc, aluminum welding system

(a)



(b)



(c)

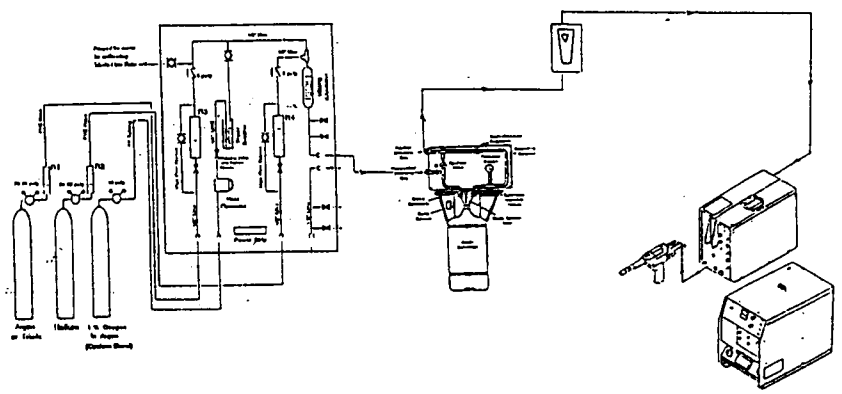


Figure 13. Gas flow line diagrams. (a) gas tungsten arc welding system (b) gas metal arc welding system (c) aluminum welding system

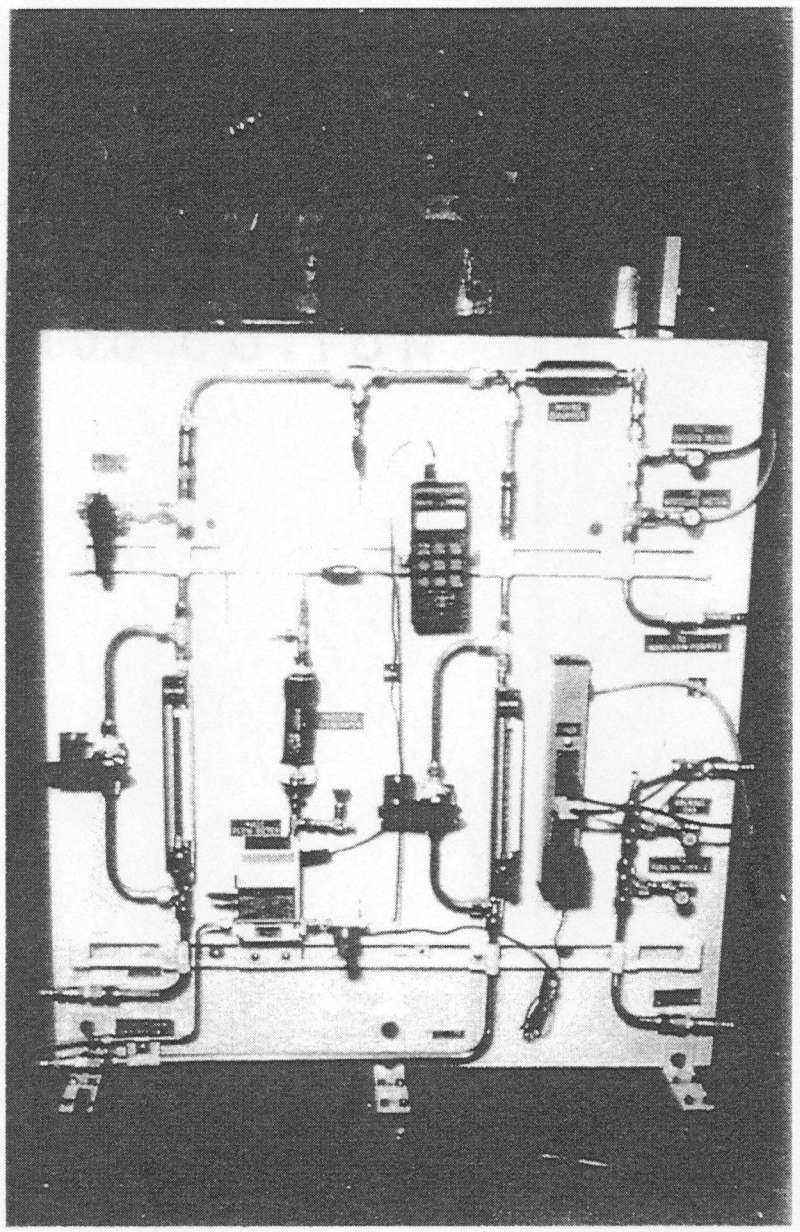
4.2. **Gas Blending Panel**

The program utilized a custom built gas-blending panel for shield gas mixing and impurity introduction. The panel was developed and fabricated by Hercules, Inc. The panel has the capability of blending argon and helium gases in ranges from 5He/95Ar to 5Ar/95He. Precise amounts of oxygen and moisture (individually, and mixed together) can be introduced into the blended gases. A maximum of 100 ppm of oxygen and 300 ppm of moisture (approximate values) can be introduced in the shield stream using the panel.

A photograph of the panel is presented in figure 14a, and a schematic of the gas-blending panel is shown in figure 14b. Argon, helium and 1% oxygen-argon cylinders are connected at the inlet of the unit. The gas pressure of the three cylinders are adjusted in a way that the gas at the lowest flow rate is at the highest pressure.

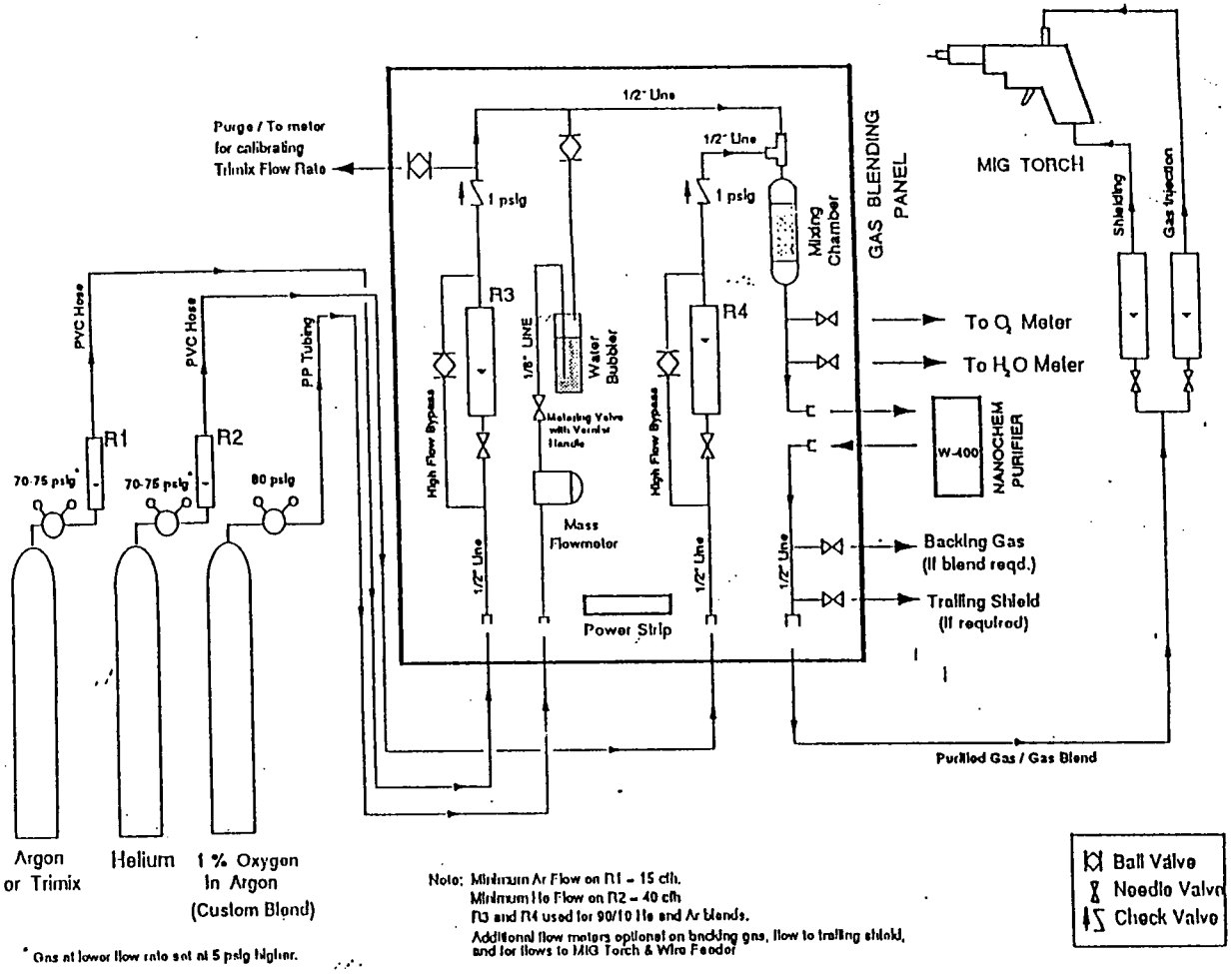
1. To obtain helium/argon mixtures in any desired ratio, the flow rates on the rotameters R3 and R4 are adjusted.
2. For preparing a gas mixture containing 5-300 ppm moisture, the 1% oxygen-argon cylinder is replaced with an argon cylinder. The water bubbler is filled to at least 3/4 capacity. The water bubbler is designed to produce a gas that is nearly saturated with moisture (>90% humidity).

The mass flow meter is calibrated to read 1-100 cc/min of air. The correction factor for the flow meter is 1.4. Hence, in order to obtain the true argon flow rate at standard conditions (70^0F at 1 atm pressure), the measured flow meter reading is multiplied by 1.4, the correction factor.



(a)

Figure 14. Shielding gas blending panel with capability to introduce moisture and oxygen in ppm amounts. (a) actual view



(b)

Figure 14 (continued). (b) line diagram

Source: Bhadha, P.M. (private communication)

3. To prepare a gas containing oxygen as the sole impurity, the water bubbler needs to be completely dried at first.

It is important to note that in practice the actual water in the gas may be lower than what is calculated theoretically. Moisture tends to form strong surface bonds on most equipment and tube surfaces. Until the entire system has been saturated with adsorbed moisture, the actual moisture concentration of the gas is lower than the calculated amount. It has been suggested by Hercules that the theoretical concentration of moisture can only be assumed equal to the actual concentration when moist gas has been flown for almost two hours. By the same token, lines that have carried moist gas must be dried by the passage of Nanochem-purified gas for at least two hours before they can be assumed to be completely dry. This second stipulation was averted by using separate lines for passage of moist gas, oxygen-contaminated gas, and Nanochem-purified shield gas. Hence, wherever possible, gas lines that had carried moisture were not used when welding with Nanochem. However, certain components of the system, as within the welding gun itself, had to be utilized to carry both moist, contaminated gas and dry, purified gas.

The system connections, hardware fittings, and the gas lines were selected to enable quick interchanges of different lines for different gases with ease. All the gas lines within the gas-blending system were made of copper. Polypropylene gas tubing (1/4" O.D.) was used to interface the different components of the system. Polypropylene has a very low permeability as opposed to the conventional rubber hoses used in gas delivery systems. Its selection thus minimized any problems associated with the permeation of air into the gas supply system.

To quanitfy the impurities injected into the gas delivery system, the procedure described in the next section was used. All argon-1%oxygen cylinders used were analyzed for exact quantification of oxygen. Wherever necessary, the individual argon and helium cylinders were analyzed for moisture and oxygen content. Because the entire gas system was leak tested under pressure prior to initiating the weld runs, leak calculations $[C/C_0 = e^{-VX/D}]$ described in the previous chapter were unnecessary.

4.2.1. **Calculation of Impurity Content in the Gas Blend**

Calculations used to determine the amount of impurities blended into the main shield gas using the gas blender were as per the verified work and results of Bhadha [57].

A = oxygen content required (ppm)

B = moisture content required (ppm)

Assume the 1% oxygen cylinder has been analyzed to have 0.95% oxygen. (95000 ppm).

T = room temperature

F = total flow rate

Total flow rate = F (cfh) = F (cu.ft/hr) X 28,216 (cc/cu.ft) X 1/60 (hr/min)

= F1 (cc/min)

'A' ppm oxygen in gas = F1 (cc/min) X A (cc) Oxygen/10^6 gas

= A1 (cc/min)

Required flow rate of 1% Oxygen blend = A1 (cc/min) X 10^6/9500 ppm

= A2 (cc/min)

Setting on mass flow meter = A2/1.4

= M

The temperature of the water in the bubbler was measured using a digital thermometer. The vapor pressure of water in mm of Hg was recorded from standard charts for the measured temperature. (P mm of Hg)

Moisture concentration in the 1% blend = [P (mm Hg)/76 (mm Hg)] X 10^6

= W1 ppm water

Moisture flow rate = A2 (cc/min) X W1/10^6

=W2 (cc/min)

Ratio of water to oxygen = 2/A1 = R

Amount of moisture in gas = (R X A) ppm

Thus, by dialing the mass flow meter at M, a gas flowing at F cfh containing A ppm oxygen and (R X A) ppm moisture could be obtained. An increase in the temperature of the water correspondingly increases the vapor pressure and humidity. Bhadha has verified injected oxygen amounts using an oxygen meter and found them to be in accordance with calculated amounts. Moisture amounts, however, are not as accurate as the values calculated theoretically using the above calculation methodology. The reason, namely adsorption of moisture on the gas lines, has already been alluded to in previous sections.

The possibility of gas sampling analysis using sample bottles was initially explored. However, this method was concluded to be inappropriate because of its inability to quantify moisture. On-line measurements using gas chromatography were deemed to be very expensive. Likewise, the use of moisture meters and oxygen meters for quantifying impurites was also ruled out

for reasons of cost. In any case, moisture meters themselves are often suspect. As mentioned, moisture tends to coat the walls of the tubing, and very long lead times, often days, can be required before moisture can desorb and the sampling lines are cleaned. During this period, the moisture analyzer would give erroneous readings. The simplest and most cost-effective solution chosen was to flow purge gas for at least two hours prior to welding. This procedure would moisten the plumbing system when welding with moisture-contaminated gas. Likewise, it would dry the adsorbed moisture when welding with pure gas. This procedure reduced any major error between the theoretically calculated amount of moisture and the actual moisture content of the gas reaching the weld pool.

4.3. **Nanochem Purifiers**

The W-400 and lab version Nanochem units were used for this program. The two purification units along with two quality flow metering devices were integrated into a single portable unit. Existing fittings were modified to incorporate Swagelok compression fittings. The unit described is depicted in figure 15.

In a program of this nature, the cleanliness aspects cannot be overemphasized. Hence, all possible sources of contamination were curtailed as far as possible. Gas lines were leak-tested with a liquid leak detector prior to each experiment. Base plates/coupons for the welding tests were mostly glass or sand-blasted, and followed by acetone cleaning prior to welding. After welding, welds were immidiately photographed prior to further handling and testing.

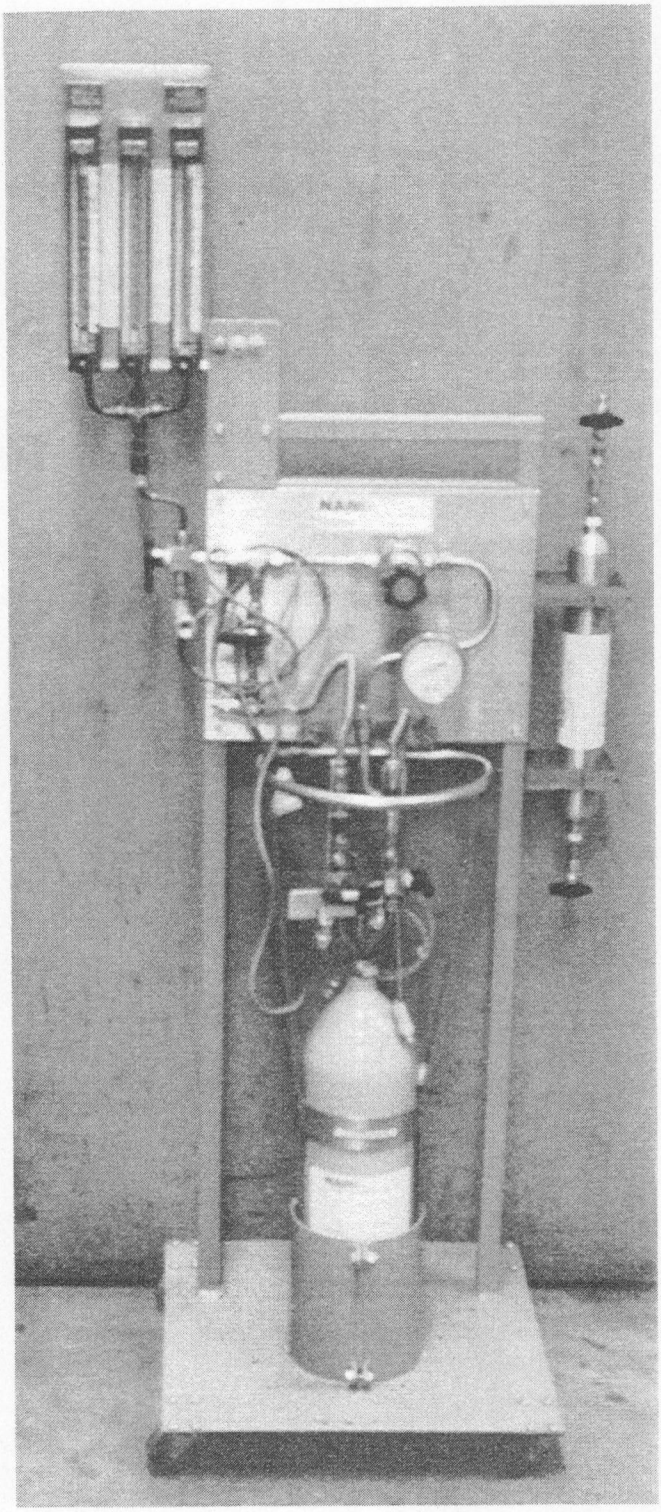


Figure 15. Modified Nanochem unit with flow meter, backing gas purifier, and castor attachments

CHAPTER 5

MATERIALS AND EXPERIMENTS

The candidate weld test materials were chosen with the calculated anticipation that they would show heightened sensitivity to the presence of impurities in the shielding gas. In addition, the selected materials were also characterized by their extensive use in industry.

The alloys used in the experiments were as follows:

1. Aluminum.-1100, 5086, 6061 series
2. Duplex stainless steel-type 255 and type 2205
3. Stainless steel-type 304 and type 316
4. AL-6XN alloy
5. HY-80 steel filler wire (100-S)
6. Titanium

The shielding gases used were as follows:

a. Argon
b. Helium
c. Argon-helium blends
d. Ar-2% oxygen
e. Ar-1% oxygen blend (used for oxygen contaminantion)

Contaminants blended into the shield gas were as follows:

a. 0-100 ppm oxygen with 10-300 ppm moisture
b. 10-300 ppm moisture

5.1. **304 and 316 Stainless Steel**

These two steels belong to the austenitic category of stainless steel. This class of stainless steels is usually regarded as the workhorse class in the stainless steel family [62]. Type 304 typically contains 19% Cr, 9% Ni, 1.5% Mn and a low 0.08% carbon (to facilitate welding by restricting carbide precipitation). Type 316 also contains added Mo (2-3%) to improve pitting resistance and has a higher creep strength. These stainless steels have the best high temperature strength and resistance to scaling and are very amenable to welding. Their corrosion resistance is also usually better than the martensitic and ferritic stainless steels. Type 304 steels are extensively used in chemical and food processing equipment. Type 316 steels are commonly used in chemical and pulp handling, photographic and food equipment.

5.1.1. **304 Stainless Steel: Autogenous GTA Weld Tests**

Autogenous GTA welds were made on 304 stainless steel plates using the following shielding gas conditions:

1. Bottled argon
2. Argon contaminated with approximately 130 ppm oxygen
2. Argon contaminated with 130 ppm oxygen and 320 ppm moisture
3. Same gas as 3, but repurified with Nanochem before use

The surface morphologies and appearances were then evaluated

5.1.2. **304 Stainless Steel: GMA Weld Tests**

The Nanochem purification unit is chemically incompatibile with gases that contain active ingredients such as oxygen and carbon-dioxide. Passing such a gas through the unit activates a hazardous chemical reaction with the resin and is not recommended. Certain alloys, however, can only be successfully welded with active components in the shielding gas. The 304 stainless steel alloy is one such alloy that is extensively welded with argon-2%oxygen shielding gas. In order to examine the possibility of using Nanochem with a fully argon shield, preliminary tests with 100% argon and argon-2% oxygen shields were conducted. The welds made with these two shields were then compared for arc characteristics and bead appearance.

5.2. **316 Stainless Steel: GTA Weld Tests**

Autogenous GTA welds were made on 304 stainless steel plates using the following shielding gas conditions:

1. Argon contaminated with 130 ppm oxygen and 320 ppm moisture.
2. Same gas as 1, but repurified with Nanochem before use.

The surface morphologies and the corrosion characteristics of the as-welded surfaces in 6% ferric chloride were evaluated using polarization procedures.

5.2.1. **Electrochemical Corrosion Test Setup**

The cyclic polarization scan tests were used to provide a qualitative view of the general anodic polarization behavior and the pitting corrosion mechanism (if any) in the test samples welded with and without purified gas. The polarization scans were used to determine the tendency of the welded alloy to undergo surface pitting or crevicing when placed in a specific environment. The scans were represented in the form of E (SHE potential) vs I (corrosion current density) plots, which were used to qualitatively asses the corrosion behavior of the test material [63].

Polarization data was generated using a computer controlled EG & G model 342 potentiostat. The test cell was used with a full capacity electrolyte/test solution (2 liters), and a silver/silver-chloride reference electrode. All solutions were prepared using distilled water. The test solutions were kept aerated by purging air through a sparger into the solution. When the solution needed to be deareated, the test solution was purged with nitrogen. All electrochemical measurements were made at 25^0C. A schematic of the cell is represented in figure 16.

Prior to initiating the polarization scan for each test, the sample was allowed to corrode freely for at least 45 minutes to allow the E_{corr} /free corrosion potential to stabilize. (The E_{corr} value depends on several factors, among which are chemical composition and metallurgical condition.) After the E_{corr} stabilized, the potential was scanned in the noble direction beginning at 25 mV below the established E_{corr} value. The scan rate used was 0.17 mV/s. The potential scan was reversed when the corrosion current density reached a

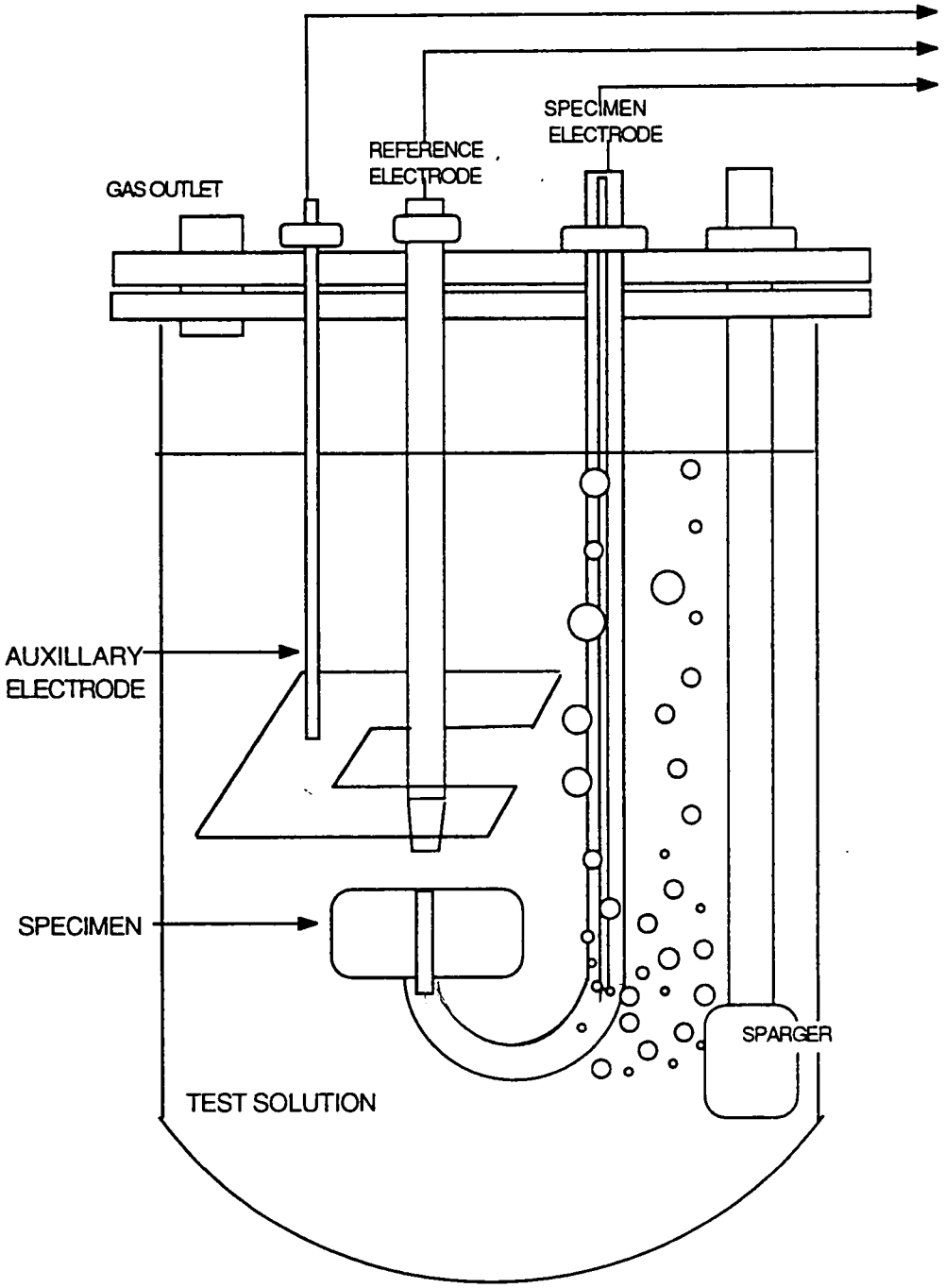


Figure 16. Corrosion test cell setup

value of 1000 uA/cm^2. The final potential on each reverse scan was programmed to be the same as the initial free corrosion potential.

The threshold current density was kept the same for all samples (1000 uA/sq.cm). This programmed threshold corrosion current density, however, does not have a bearing on initiating the reverse scan because the controlling parameter is essentially the vertex potential. The scan only reverses upon reaching the vertex potential, irrespective of the threshold current.

5.3. **Titanium**

Pure titanium is a sliver-colored metal that melts at approximately 3035^{0}F and has a density of 4.51 g/cc [64]. It has a great chemical affinity for oxygen and tends to form a tight microscopic film of oxide on freshly prepared surfaces even at room temperature. This tendency bestows a natural passivity to further reaction in other media, which accounts in part for its excellent corrosion resistance. Titanium and its alloys are commercially available in plate, sheet, bar, forging, extrusion, pipe, and tube forms. Because of its light weight, excellent corrosion resistance, and high strength to weight ratio, its applications in chemical, marine, aviation, space, and military fields are quite extensive. During welding, it has the propensity for dissolving discrete amounts of its own oxide in solution. Oxygen and nitrogen taken into solution usually strengthen and embrittle titanium welds. Hydrogen also has a definite embrittling effect. Hence, highly inert atmospheres are required during welding.

5.3.1. **GTA Autogenous Weld Tests: Titanium Welds**

Autogenous GTA welds were made on pure titanium plate using three conditions of the shielding gas.

1. Bottled helium
2. Helium contaminated with moisture (approximately 300 ppm)
3. Gas indicated in 2, but Nanochem-purified before use

5.4. **Aluminum 1100**

This alloy is usually 99% or higher purity aluminum [65]. It has widespread applications especially in the chemical and electrical fields. The alloy is characterized by a high corrosion resistance, high thermal and electrical conductivity, low mechanical properties and excellent workability. Moderate increases in strength are usually obtained by strain hardening. Iron and silicon are the major impurities. Some typical applications of this material include plumbing fixtures, piping, deep drawn products, spun hollow ware, furniture, kitchen equipment, and decorative/architectural applications.

5.4.1. **Aluminum 1100: Autogenous GTA Weld Tests**

Autogenous GTA welds were made on 1100 aluminum using the following gas conditions:

1. Bottled helium
2. Helium contaminated with 300 ppm moisture

3. Same gas as 2, but repurified before supply to the weld torch

5.4.2. **1100 Aluminum: GMA Weld Tests**

Several initial weld trial runs were carried out using bottled argon to establish the optimum welding parameters for welding in the spray mode. The final test parameters selected were as follows:

Current: 230-260 amps

Voltage: 24 voltages

Travel speed: 45 ipm

Shield gas: Bottled argon

Flow rate: 45 cfh

Electrode stick out: 0.6"

Prepurge: 10 seconds

Postpurge: 2 seconds

Bead-on-plate welds were then made on 1100 aluminum (8" X 2" X 0.15"coupon size) with 1100 filler (1/16") using the following shielding gas conditions:

1. Argon gas contaminated with varying amounts of moisture (11-375 ppm) and oxygen (5-192 ppm)
2. Same as 1, but repurified with Nanochem before use

Representative samples exhibiting the difference between coupons welded with the bottled gas, the contaminated gas, and the repurified gas were studied. The studies encompassed the following aspects:

1. Surface appearance
2. Metal transfer characteristics using arc sound data
3. Bend ductility

5.5. **Aluminum 5086**

This is a moderate strength, non-heat treatable alloy using magnesium as its principal alloying element. The alloy possess good welding characteristics and good resistance to corrosion in marine atmospheres. Certain limitations are placed on the amount of cold work and safe operating temperatures permissible to avoid susceptibility to stress corrosion. Typical applications include marine components, tanks, tankers, truck frames.

5.5.1. **GMA Weld Tests : 5086 Aluminum**

Bead-on-plate welds were made on 5086 aluminum with 5556 filler wire using the following shielding gases:

1. 65% argon-35% helium contaminated with oxygen (100 ppm) and moisture (300 ppm)
2. Same gas as 1, but repurified with Nanochem prior to use
3. Helium contaminated with approximately 300 ppm moisture
4. Same gas as 3, but repurified with Nanochem prior to use

The ranges of these contaminant levels were selected to be reflective of contaminant levels often found in welding grade cylinders in the as-bottled state. Also, minor leaks in the system plumbing often cause contaminants to

ingress in quantities higher than even those selected for this experiment. Helium was also added to the list of test gases (only argon was used in the previous GMA tests with 1100 aluminum) because helium is usually a better shield than argon for welding aluminum. It is more favored because of its relatively high ionization potential that causes it to run hotter than argon, thus putting more heat into the weld. The additional heat is beneficial owing to the high conductivity of aluminum. The welding parameters employed were as follows:

Current: 150 amps

Voltage: 19 V

Wire feed speed: 3.2 (dial reading)

Travel speed: 16 ipm

Flow rate: 50 cfh

The choice of this particular filler-parent combination for shielding gas purity effects assessment was influenced by its specific relevance to a situation where gas contamination and its effects are a practical occurrence. The Navy utilizes the 5086 plate -5556 filler combination for welding marine structures. Also, a dockyard welding facility represents a typical case of GMA welding equipment susceptible to shielding gas contamination. Long lengths of tubing, manifold distribution systems, and a larger number of fittings in the gas delivery system contribute towards increasing the probability of leaks leading to contamination of the shielding gas. In view of these factors, investigation of the potential of Nanochem purification was considered worthwhile.

5.6. **Aluminum 6061**

This alloy contains silicon and magnesium in approximate proportions to form magnesium silicide and is thus capable of being heat treated. It is one of the most versatile in the 6000 series of aluminum alloys and possess good formability, corrosion resistance and medium strength. The alloy is usually formed in the -T4 temper (solution heat treated but not artificially aged) and then reaches full -T6 properties by artificial ageing. Some typical applications include pipe and pipe fittings, structural and architectural parts, automotive components, railway, and marine parts.

5.6.1. **6061 Aluminum: GMA Weld Tests**

In the experiments conducted earlier using GMA and GTA processes for welding 1100 and 5086 aluminum, it was found that a clear distinction exists when welding aluminum with and without purification of contaminated shielding gas. It was decided to replicate the experiments using 6061-4043 aluminum, which is essentially the workhorse of all structural applications using aluminum. It was also decided to refine the test procedures when conducting this round of tests by performing the following:

1. Using a filler wire size (0.045") that is more relevant to practical applications, as opposed to the 1/16" fillers used in the previous tests.

2. Using parameters that produce welds more realistic in appearance with flatter, wider beads as opposed to the slightly "bumpy" beads produced when welding with the 5556 wire in the previous experiments.
3. Using mechanical testing exactly as prescribed by ASME boiler and pressure vessel codes. Hence, base plate coupons measuring 3/8" X 6" X 1-1/2" were used for the tests, which could be used after minimum machining as bend test samples for subsequent mechanical testing.

Before initiating the actual tests, careful attention was first paid to machine setting to produce good metal transfer characteristics when welding and excellent bead appearance after welding. Since tests conducted earlier revealed that helium seemed more susceptible to the introduction of moisture in terms of producing degrading effects in the weld, the current experiments were carried out using helium. (As alluded to before, oxygen may have the effect of actually reducing the amount of porosity in aluminum welds.) The gases used for the tests were as follows:

Bottled helium

Bottled helium contaminated with approx. 300 ppm moisture

Same gas as above but Nanochem-purified before use

The other welding parameters used were as follows:

Current: 190-200 Amps

Voltage: 26 V

Electrode stick-out: 1/4"

Gas flow rate: 45 cfh

Travel speed: 18 ipm

5.7. **AL-6XN Alloy**

The AL-6XN alloy (UNS N08367) is a low carbon, high purity super austenitic stainless steel with high nickel (25%), molybdenum (6.5%), and nitrogen (0.2%) [66]. The alloy is an extension of the AL-6X alloy and provides good resistance to a broad range of very corrosive environments. Its high strength and corrosion resistance make it a better choice than the conventional duplex stainless steels and a cost effective alternative to the more expensive nickel-base alloys in applications where excellent formability, weldability, strength, and corrosion resistance are essential. The nitrogen and molybdenum content of the alloy serves to further increase the pitting and crevice corrosion resistance, improve resistance to localized corrosion in oxidizing chlorides and reducing solutions, and increase the strength of the alloy. The nitrogen also reduces the potential for sigma phase formation during welding, especially in thicker sections, where even small amounts of sigma have been found to cause a degradation of properties.

The AL-6XN alloy is well suited for important industrial applications such as:

Condensers, heat exchangers, and piping containing sea water

Chemical process tanks and pipelines

Power plant flue gas scrubber environments

Oil distillation columns

Service water piping systems for nuclear power plants

5.7.1. **Welding of AL-6XN**

The low carbon and high nitrogen content make AL-6XN readily weldable in all commercially available thicknesses. The normal welding practices used for austenitic stainless steel can be applied. Emphasis, however, should be placed on cleanliness to avoid oxygen and carbon contamination. Sheet material is amenable to autogenous GTA welding. Nitrogen at 3% to 5% can be added to the argon shielding gas where service in severe chloride environments is anticipated. For tube sheets of type 316 stainless steel or higher alloy material, seal welds without filler wire are adequate. No loss of corrosion resistance in the weld or heat-affected zone owing to the presence of carbides or sigma phase has been found in experimental studies. In the case of heavier plate welding, Hastealloy C22 or Inconel 625 filler metal is generally used to provide a good corrosion resistance match for the alloy. Manual GTAW is the preferred welding method with emphasis on good edge preparation and fit-up, followed by acetone cleaning. It is recommended for removal of any imperfections formed after the arc has been extinguished during multipass welding, by back-grinding with aluminum oxide wheels. Changes in the flow characteristics of the molten metal may be indications of flaws, and these areas should be ground and rewelded. Good welding technique is important in order to avoid oxide inclusions and overheating of the workpiece. Welding using a shielding gas of 75% helium-25% argon or pure argon for the root pass usually gives the best results.

5.7.2. **AL-6XN : GTA Autogenous Spot Weld Tests**

The AL-6XN alloy was a fitting choice in the experiments to research the effects of shield gas purity on weld behavior. The reasons for selecting the AL-6XN alloy as a candidate material were as follows:

1. The growing importance of AL-6XN, as attested by the applications discussed in the prior sections
2. Its promise as a relatively lesser researched alloy with high utilization potential
3. Its high purity nature, which makes it susceptible to impurities during welding

Gas tungsten arc spot welds were made on AL-6XN sheet coupons using different levels of contaminants in the argon shielding gas. The rationale behind selecting this welding method was as follows:

1. Microsegregation in GTA autogenous welds causes regions to be more susceptible to localized corrosion.
2. For spot welds made with contaminated shielding gas, the center of the weld/crater essentially contains the most contamination. This fact was repeatedly determined in previous bead-on-plate GTA trials using 304 type stainless steel and 255 duplex stainless steel. The phenomenon is easily explained because the contaminants in the molten puddle are swept along with the moving arc until the arc is extinguished at the weld stop. Thereafter, the dissolved gases escape or remain within the solidifying matrix of the weld crater. A spot weld can be looked upon as a weld stop per se. Hence, a spot weld made with impure shielding gas will contain all the aggregated impurities that are otherwise retained in the weld crater of an actual weld.

3. It was endeavored to examine the feasibility and also develop procedures for a spot weld test methodology to be used as a quick, inexpensive, and effective method for assessing gas purity prior to welding start-up in a production environment.
4. Moreover, spot test coupons offer the added convenience of being amenable to subsequent tests such as corrosion tests, SEM examination, and routine metallographic testing.

Twenty five spot weld test coupons (1"x1"X 0.05") were prepared from a 0.32" thick sheet of AL-6XN. Considering the nature of this study, special attention was paid to cleanliness and especially to the prevention of contamination arising during handling. The coupons were degreased with acetone and oven-dried prior to welding. Specimens were handled with surgical gloves. The gas cups, gas screens, and tungsten electrodes were also acetone cleaned and dried. All system fittings were leak-tested with a liquid leak detector. The welds with and without purification were made in two separate batches.

Backing gas was also provided to shield the molten underside/root of each weld. A smaller capacity Nanochem unit (lab version) was used to purify the backing gas. Shield gas contamination was achieved by employing the gas blending panel. Approximately 1-300 ppm moisture and 1-100 ppm oxygen were used in the tests.

The following three gases were used for conducting the GTA weld tests:

1. Bottled argon
2. Bottled argon contaminated with oxygen and moisture

3. Bottled argon contaminated with oxygen and moisture but Nanochem-purified before use

The parameters employed for the spot welds were as follows:

Current: 85 amps

Tip to work distance: 3 mm

Arc on time: 6 seconds

Arc decay: 1.5 seconds

Polarity: DCSP

Shield gas: Welding grade argon

Flow rate: 18 cfh

Backing gas: Welding grade argon

Flow rate : 4 cfh

Electrode: 2% thoriated, 3 mm diameter tungsten

The system lines were purged for 1.5 hours before welding to ensure that moisture concentrations calculated were equal to the actual amounts present in the gas. Moisture forms strong bonds with system plumbing, and an equilibrium must be established between the gas and the inside walls of the plumbing system for the calculated moisture in the gas to be equal to the actual content.

The following aspects were examined to evaluate and compare the coupons welded with purification and with contaminated gas:

1. Surface appearance using stereo microscopy
2. Surface characterization using high resolution scanning electron microscopy
3. Metallography and hardness

4. Electrochemical corrosion tests of the fusion zone (as-welded and ground conditions, heat-tinted area, entire weldment) in the following industry-relevant acidic and basic media:

    6% ferric chloride

    10% sulphuric acid

    50% boiling sodium hydroxide

5. Immersion corrosion tests in 50^0C ferric chloride

5.8. **100-S Filler Wire (for HY-80 Steel)**

The HY-80 ultra service steel is a weldable, high strength, quenched and tempered steel made to the restrictive practices common to the production of armor plate steels. HY stands for high yield strength 80 ksi. min. Their superior notch toughness and ductility at this strength level recommend them for applications subject to dynamic loading and/or low temperature service.

This steel was initially included in the program because notch toughness and cold cracking susceptibility, of special relevance to the applications of these steels, are both factors that can be significantly affected by the degrading effects of moisture and other shield gas impurities. Some of the typical applications of HY-80 steels are listed below:

Pressure hulls in US Navy submarines

Hulls of US Navy carriers, cruisers, destroyers, frigates

Machinery and equipment shock test platforms

Automotive assembly

Welding high strength steels using a gas shielded welding system containing defective hoses, contaminated cylinders, and leaky fittings constitutes an ideal situation wherein the weld is susceptible to the degrading effects of moisture. Likewise, it is also the ideal situation for the deployment of gas purification systems such as Nanochem.

Accordingly, it was decided to investigate the effects of moisture and oxygen on the toughness and diffusible hydrogen content of high strength GMA welds, and any possible benefit derivable from the use of Nanochem. The diffusible hydrogen content was decided to be investigated because moisture introduced into the weld pool through defective hoses, improper fittings, and bad cylinders is capable of dissociating into hydrogen, which can increase cold cracking susceptibility.

100-s filler wire was procured with the initial idea of GMA welding full penetration HY-80 steel weldments for Battele cold cracking tests and mechanical properties tests. The 100-S wire chemistry used is as follows:

[.08% C (max), 1.25-1.80 Mn, .20-.55% Ni, .25-.55% Mo]

The 100-S wire is designed to produce weld deposits with a minimum 82,000 psi yield strength with excellent impact properties even when welding at high heat inputs (up to 110,000 J/in). Shielding gases used when welding with this wire are 98% argon-2% oxygen, or 95% argon-5% carbon dioxide [67]. (The U.S. Navy uses 94% argon-6% carbon dioxide.)

5.8.1. **100-S Filler Wire: GMA Weld Tests**

The initial GMAW tests were carried out using two different shielding gases:

1. Argon-2% oxygen
2. Argon

The parameters used for the welding were as follows:

Current: 180 Amps

Voltage: 24

Travel speed: 16 ipm

Gas Flow rate: 40 cfh

It was necessary to conduct this preliminary test in order to ascertain exactly how much of an effect the active shield gas component (2% oxygen) had on the process characteristics and bead appearance. (The recommended shield gas for the 100-S filler wire is Ar-2%O_2, or Ar-6% CO_2, or the Navy recommended Ar-6%CO_2.) In other words, the utilization of an active additive in the base shield mixture is deemed necessary, thus restricting the deployment of Nanochem.

Earlier experiments had indicated that welding stainless steel with argon-2%oxygen, after passing it through the purifier, resulted in poorer arc characteristics and weld bead shape. This result was attributed to the filtering effect caused by the Nanochem unit which resulted in the removal of the oxygen component from the main argon shield gas. The resulting pure argon is not suitable for GMA welding stainless steel.

Lest similar effects be produced in the proposed experiments with the GMA welding of the HY steels, the feasibility of welding with pure argon was first evaluated with the two weld tests described above. Although the gases used were different, the parameters used were identical. This was deemed necessary to bring out the exact effect the shielding gas produced in the arc characteristics and the bead appearance.

5.8.2. **100-S Filler Wire: GTA Weld Tests**

Unlike the GMA process for welding the HY steels, where active components are a recommended feature of the gas shields used, the GTA process inherently prohibits the use of active gases. This prevents degradation of the tungsten electrode used in the process. In other words, the Nanochem unit could be easily used for purification of the inert gas used in the GTA process. This is in contrast to the GMA process, where using the Nanochem unit is not only not feasible due to chemical incompatibility with the active 2% oxygen component in the shield gas, but also unsafe.

The aim of the GTA experiments was to determine the effect of moisture and oxygen contamination in the shield gas and quantify the effect in terms of diffusible hydrogen content. The diffusible hydrogen in a weld, acting in concert with stress and a susceptible martensitic microstructure (35 HRC) can lead to cold cracking. Since moisture in the gas shield dissociates within the heat of the arc and forms hydrogen and oxygen, the importance of gas purity cannot be overemphasized.

The moisture and oxygen contaminants (approximately 300 ppm and 100 ppm, respectively) were introduced into the helium shield gas using the gas blender. Three coupons were welded with the contaminated gas and three were welded after Nanochem purification. The effect of this varied shield gas purity was determined by measuring the diffusible hydrogen, the details of which are described in the subsequent section. For two coupons, (welded with and without subsequent purification), the gas lines were also coated with moisture prior to initiating the welds.

The parameters used for the automatic GTA welding were as follows:

Current: 200 amps

Voltage: 14 volts

Speed: 8 ipm

Gas flow rate: 20 cfh

Filler wire diameter: 0.045"

5.8.2.1. **Diffusible Hydrogen Test Procedures**

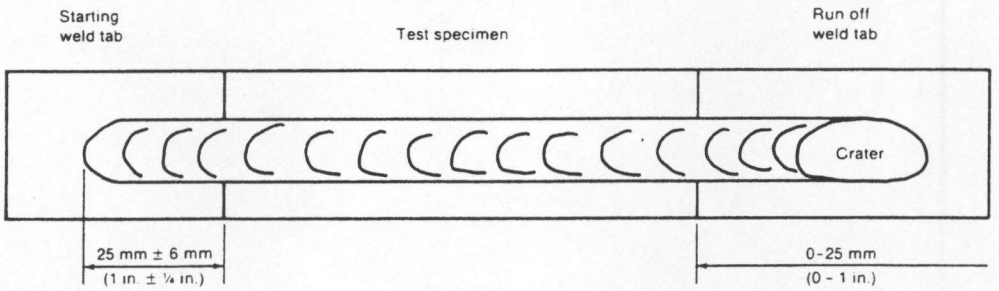
The AWS A4.3-86 "Standard Method for Determination of the Diffusible Hydrogen Content of Martensitic, Bainitic, and Ferritic Weld Metal Produced by Arc Welding" was followed to determine the diffusible hydrogen content in the 100-S filler wire weld deposits made using the GTA process, using purified and contaminated gas. The test specimens taken from A-36 bar stock were sectioned into 31/8" X 1" X 1/2" in. coupons. The specimens were then degassed at 450^0C for three hours and the scale removed by dry grinding. The samples were then cleaned with acetone and weighed on an electronic

balance. The samples were held in a special copper fixture along with 1 in. long run-off tabs.

The arc starts and stops during welding were selected to be carefully contained only on the center of the weld tabs (figure 17a). After welding, each weld test assembly was quickly removed from the copper fixture and plunged into ice water within 5 seconds of extinguishing the arc. The weld test assembly was then agitated in the ice water and quickly transfered to a liquid nitrogen bath. After about two minutes in the liquid nitrogen bath, the test assembly was removed from the bath and the tabs were broken by a hammer impact on the un-welded side. The test specimen was then once again immersed in the liquid nitrogen bath. For each condition of the shielded gas (purified and contaminated), three coupons were welded. For the fourth coupon welded in each of the categories, the main gas line was first saturated with moisture (i.e. the line to the torch when purification was not used, and the line to the Nanochem unit when purification was used). The line was saturated by passing a stream of water through it for two seconds and then draining away all visible drops of water.

At no time was the welded specimen allowed to stay out of the liquid nitrogen bath for more than a minute. No wire brush cleaning was employed before transfering the specimens to the sampler for collecting the diffusible hydrogen sampler. After this point, the individual sample chambers were purged with high purity argon gas at 1.5 bar pressure for 2 minutes each. The sampler unit was then placed in an oven maintained at 45^0C for 72 hours. Usually, the hydrogen analysis is carried out at the end of 72 hours after the hydrogen extraction cycle. However, owing to gas chromatograph equipment

(a)



(b)

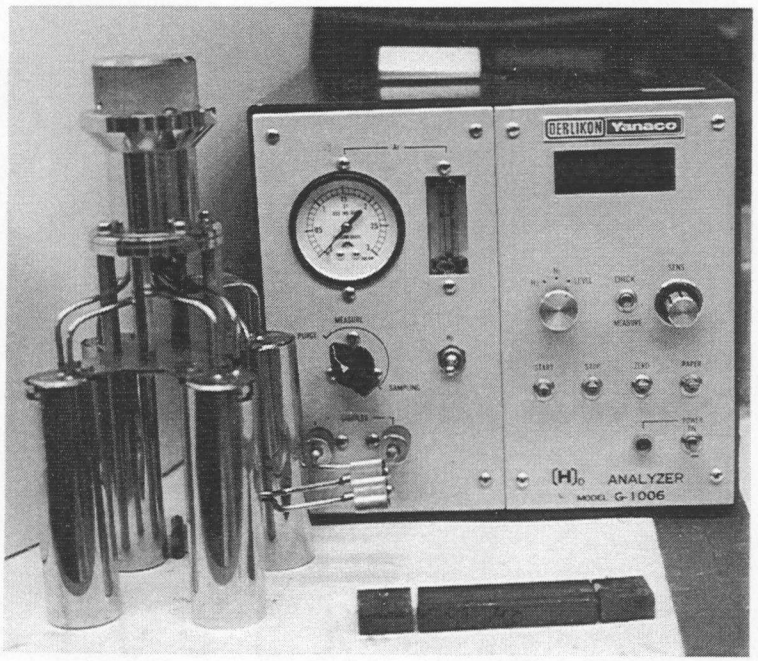


Figure 17. Diffusible hydrogen measurement equipment. (a) weld test coupon (b) diffusible hydrogen measurement chromatograph

breakdown, the sampler unit (figure 17b) had to be shipped to Hobart Bros. Ohio on two different occasions. The analysis (at corrected standard temperature and pressure) provided the volume (ml/100g weld metal) of hydrogen contained in each weld. After the analysis, the samples were weighed again and the diffusible hydrogen calculated in ml/100g of deposited 100-S filler weld metal.

5.9. **Duplex Stainless Steel: 255 and 2205**

The family of duplex stainless steels is an example of the versatility of steel. In this age of new materials, these stainless steels show that steel is a very competitive material with a great potential for development. Compared to the original 13% chromium steel invented almost a century ago, duplex stainless steels are a new brand of materials, about as new as ceramics, composites, and polymers.

Duplex, a one part ferritic and the other part austenitic steel, provides the best of both worlds in terms of properties [68]. The ferrite provides strength and resistance to stress-corrosion cracking, while the austenite provides good toughness and corrosion resistance.

The development of these steels was propelled by the need for industrial materials that found applications in chloride ion containing aqueous fluids, requiring both corrosion and/or strength properties. The development carried out is manifested in today's new duplexes, which are low carbon steel grades with 22-27% Cr, 4-8% Ni, 0.1-0.3% N (with or without 1.5-4 % Mo), and in some cases additional elements.

The corrosion behavior of these steels is characterized by their commendable chloride stress-corrosion resistance and high resistance to pitting corrosion in more severe conditions. These properties can be coupled with 100% higher proof strength properties as compared to austenitic stainless steel grade AISI 316-L in a temperature range of -10^{0}C to + 250^{0}C. Duplex steels are almost exclusively produced by the AOD steel-making method, in conjunction with electric arc melting processes.

The line of demarkation between Super DSS and DSS is not well defined. Super duplex stainless steel would be associated with about 25% Cr, >3.5% Mo and >0.2% N, providing an increased Pitting Resistance Equivalent (PRE) where PRE = (%Cr+ 3.3 X %Mo + 16 X % N) > 40.
The regular Mo-alloyed duplex grades have a PRE value between 30 and 36. It has been a gradual process, but the appreciation for duplex stainless steels is finally coming in. Designers of components for the oil, gas, chemical, utility and paper-making industries are all identifying niches and applications for these relatively new materials.

5.9.1. **Filler-Added GTA Weld Tests: DSS 255**

Two GTA welds were made on 255 duplex stainless steel using 255 solid wire filler (0.035"). Two conditions of gases were used:

1. Argon contaminated with 130 ppm oxygen and 300 ppm moisture
2. Argon contaminated with 130 ppm oxygen and 300 ppm moisture
3. Same gas as 3 above, but Nanochem-purified before supply to the torch (No filler wire was added for the welds made with this gas)

5.9.2. **2205 Duplex Stainless Steel: GMA Weld Tests**

The tests conducted by Hercules, Inc., on GTA-welded Ferralium and E-Brite had revealed the potential for improvement in weld mechanical properties using Nanochem-purified gas. In order to extend the scope to gas metal arc welding and investigate the effects on more popular alloys, the duplex 2205 stainless steel alloy was chosen for the welding tests.

Preliminary bead-on-plate trials for assessing the optimum weld test parameters were carried out. The shielding gases assessed were welding grade argon, argon-2% oxygen, and welding grade helium. The overall arc characteristics of the weld shielded with argon-2% oxygen were the best. The wettability of the welds made with pure argon was reduced visibly. However, since the utilization of Nanochem restricts the use of shielding gases with oxygen components, the use of the industry-recommended argon-2%oxygen shield was ruled out.

Five weld test assemblies (5" X 5" X 1") were prepared for welding. Run-off tabs were welded to the ends of each assembly to accomodate the weld starts and weld stops, ensuring full arc stability in the groove. Welding parameters selected were as follows:

Current: 210-220 amps

Voltage: 25-26 volts

Travel speed : 12- 14 ipm

Gas flow rate: 45 cfh

Electrode stick out: 1/2".

Interpass temperature was maintained at approximately 60-100^{0}F. Approximately 20 passes were required to fill the groove of each test assembly. Careful attention was paid to the cleaning of the weld groove with appropriate grinding and solvent cleaning procedures.

The gases used for welding the five assemblies were as follows:

Argon-2% oxygen

Bottled argon

Argon contaminated with moisture

Bottled argon purified with Nanochem

Argon contaminated with moisture and purified with Nanochem

The assemblies welded with the five different gases/gas conditions were sectioned for test specimen extraction. Figure 18 depicts the locations from where the samples for bend, toughness, corrosion, and microstructural evaluation were extracted. Charpy impact test specimens, corrosion specimens, bend specimens, and metallography specimens were extracted and evaluated.

5.10. **GMA Torch Gas-Injection Experiments**

Necessary torch modifications were performed to incorporate gas-injection into the Miller GA-50A torch in accordance with the original patent description. Following these modifications to pressurize the liner with inert gas, extensive gas flow and gas pressure measurements were taken at the wire inlet

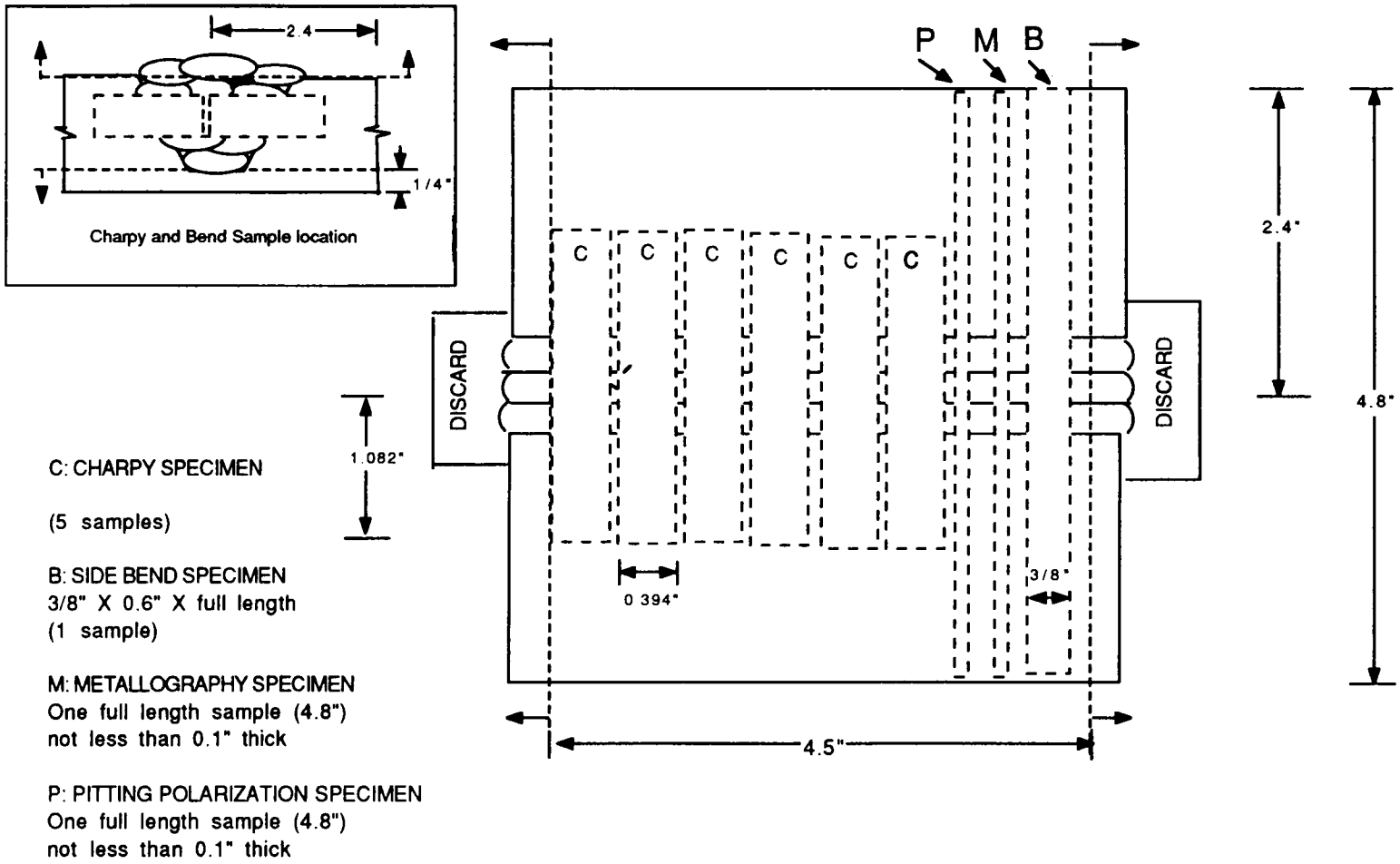


Figure 18. GMA-welded 2205 duplex stainless steel assembly showing sample extraction plan

and the contact tip ends of the wire feed cable. These measurements were based on three different gas shielding configurations, as follows:

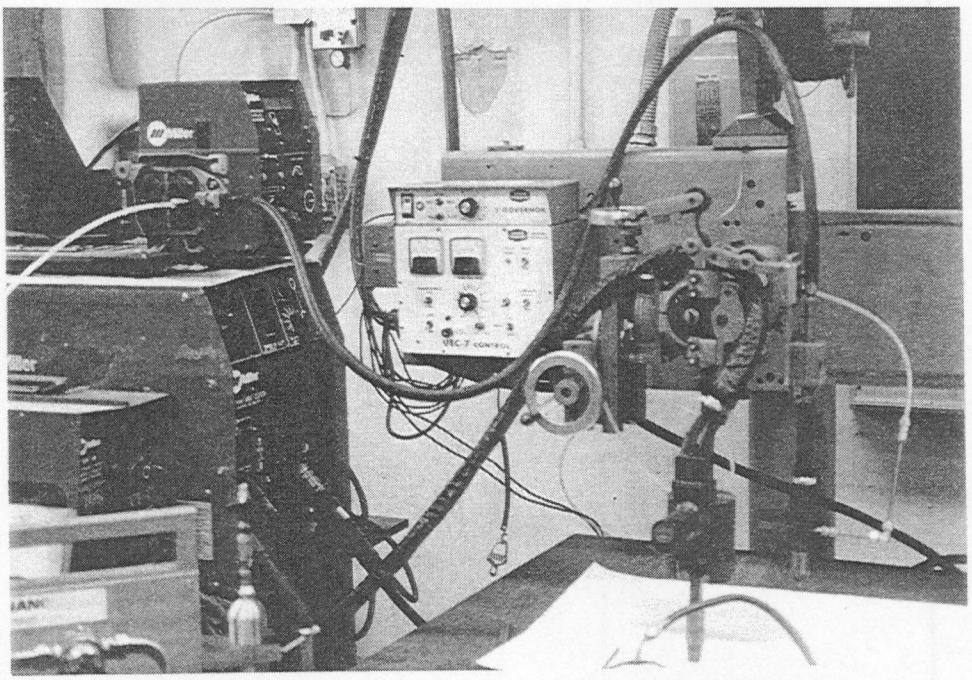
1. Regular configuration gas shielding without gas injection
2. Modified configuration using gas-injection modification as per described patent (injection into the liner)
3. Dual injection- One injector to pressurize the wire inlet adapter; one injector to pressurize the contact tip area (figure 19)

The flow rates and flow pressures at the contact tip and the cable inlet ends were measured using a bubble flow meter and a water U-tube manometer. The analytical instruments were interfaced with the wire cable (figure 20) using appropriate sections of tygon tubing. Test welds were also made on 304 stainless steel plate using each of the three configurations above to check for differences in arc characteristics and weld bead appearance.

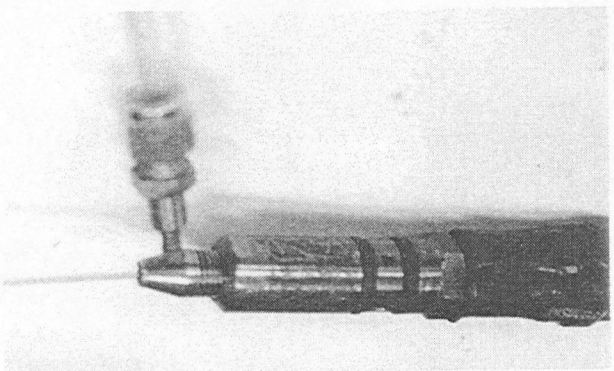
5.11. **GTA Gas Injection Experiments**

The wire feed nozzle of the automatic GTA torch was modified to allow pressurization of the wire feed tube to generate positive gas flows at the wire feed tube entry and exit ends. After these modifications to prevent air entrainment into the wire feed tube and weld pool were performed, GTA bead-on-plate welds were made on 304L stainless steel. Nanochem-purified argon shielding gas was used in the following two gas shielding configurations:

1. Regular shielding configuration without gas injection
2. Regular shielding configuration with 2 cfh gas injection



(a)



(b)

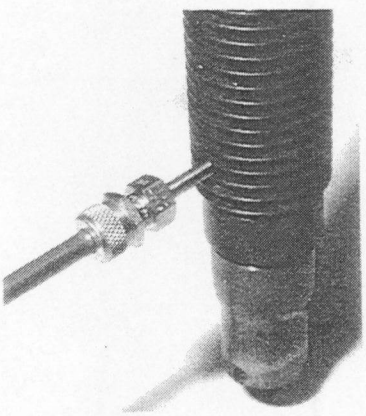


Figure 19. Dual-injection in GMA torch cable showing location of two gas injectors to prevent air entrainment. (a) wire inlet (b) wire outlet.

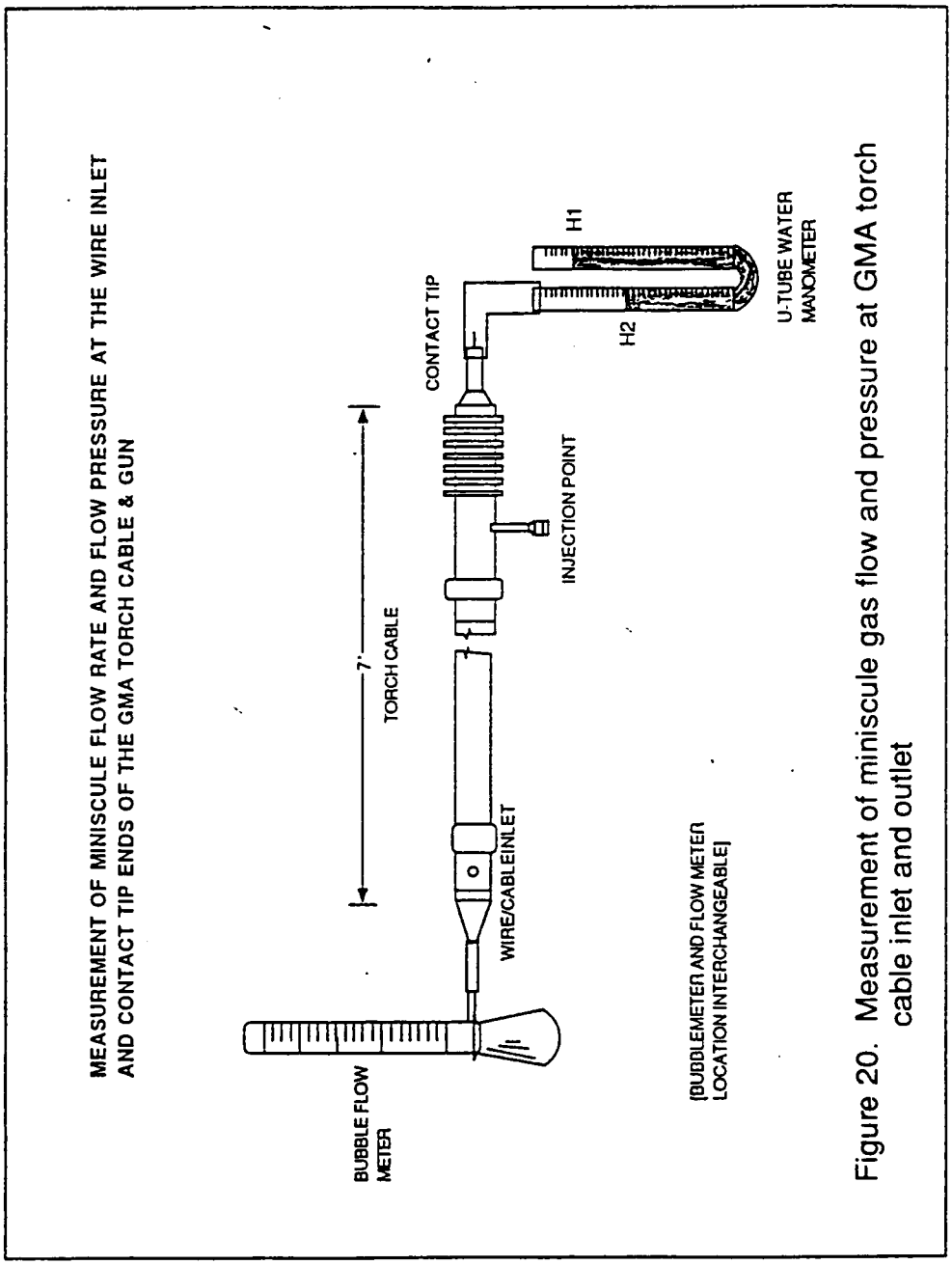


Figure 20. Measurement of miniscule gas flow and pressure at GMA torch cable inlet and outlet

CHAPTER 6

RESULTS AND DISCUSSIONS

6.1. 304 Stainless Steel: Autogenous GTA Weld Tests

The effect of shielding gas impurities on autogenous GTA welds of 304 stainless steel was perceivably detrimental. This fact is attested by figure 21a, which indicates the surface of the 304 GTA weld made with approximately 320 ppm moisture and 130 ppm oxygen present in the argon shield gas. The surface appears severely degraded and almost totally black. When only oxygen was added to the shield stream, the effect is still deleterious to the appearance of the weld as attested by its oxidized appearance depicted in figure 21b. The degree of oxidation is, however, not as gross as the case when in addition to the oxygen, 300 ppm of moisture was also added to the shield stream.

In contrast, when moisture-and-oxygen contaminated gas is purified with Nanochem, a clean, shiny, and acceptable weld is produced, as shown in figure 21c. The sample welded with the bottled argon (without any added contamination or re-purification) appeared similar to the weld made with Nanochem purification in that the weld was shiny and smooth with no evidence of oxidation. (A comparison of weld made with welding grade gas and with Nanochem-purified gas is presented in figure 22.) This result suggests that bottled gas used for welding was of sufficiently high purity. Such high purity and consistency may not necessarily always be the case in a regular field

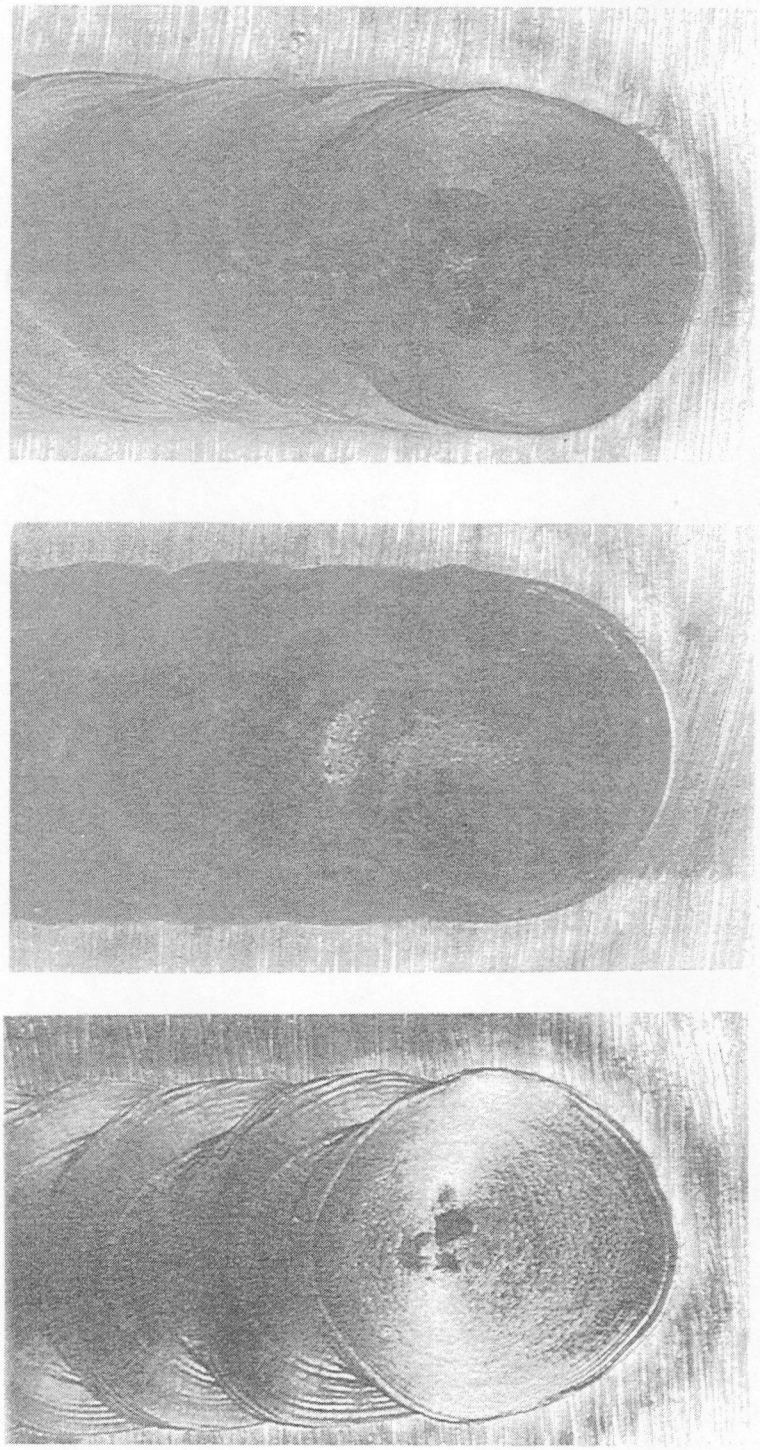


Figure 21. Effect of shield gas purification on the surface appearance of 304 stainless steel, GTA autogenous welds. (a) 132 ppm oxygen in argon shield (b) 132 ppm oxygen, 320 ppm moisture in argon shield (c) Nanochem purified argon shield originally containing 132 ppm oxygen and 320 ppm moisture



Figure 22. Comparison of weld appearance of 304 GTA autogenous welds using (a) welding grade argon shield and (b) Nanochem-purified, originally contaminated argon shield

situation. Hence, Nanochem purification is recommended to offset the effects of inconsistent bottled gas purity.

6.2. **304 Stainless Steel: GMA Weld Tests**

The typical appearance of a GMA weld shielded with argon-2%oxygen is indicated in figure 23a. The bead is smooth and indicates good wettability. The arc characteristics were also noted to be good. In contrast, the appearance of a GMA weld shielded with 100% argon shielding gas passed through the Nanochem unit shows poor bead appearance and wettability. The transfer mode was also more globular and associated with visible spatter (figure 23b). Evidently, the use of argon-2%oxygen gas is best suited for GMA welding the 304 stainless steels. Therefore, the use of 100% argon shield, and likewise, the deployment of Nanochem for GMA welding the 304 stainless steels, was not considered in any further tests.

6.3. **316 Stainless Steel: Autogenous GTA Weld Tests**

6.3.1. **Surface Appearance**

Similar to the behavior exhibited by the 304 GTA autogenous welds, the 316 GTA autogenously welded samples also indicated significant differences between welds shielded with contaminated gas and welds shielded with Nanochem-purified gas. Figure 24a depicts the appearance of a weld shielded with moisture-and-oxygen contaminated argon. The arc wander was significant

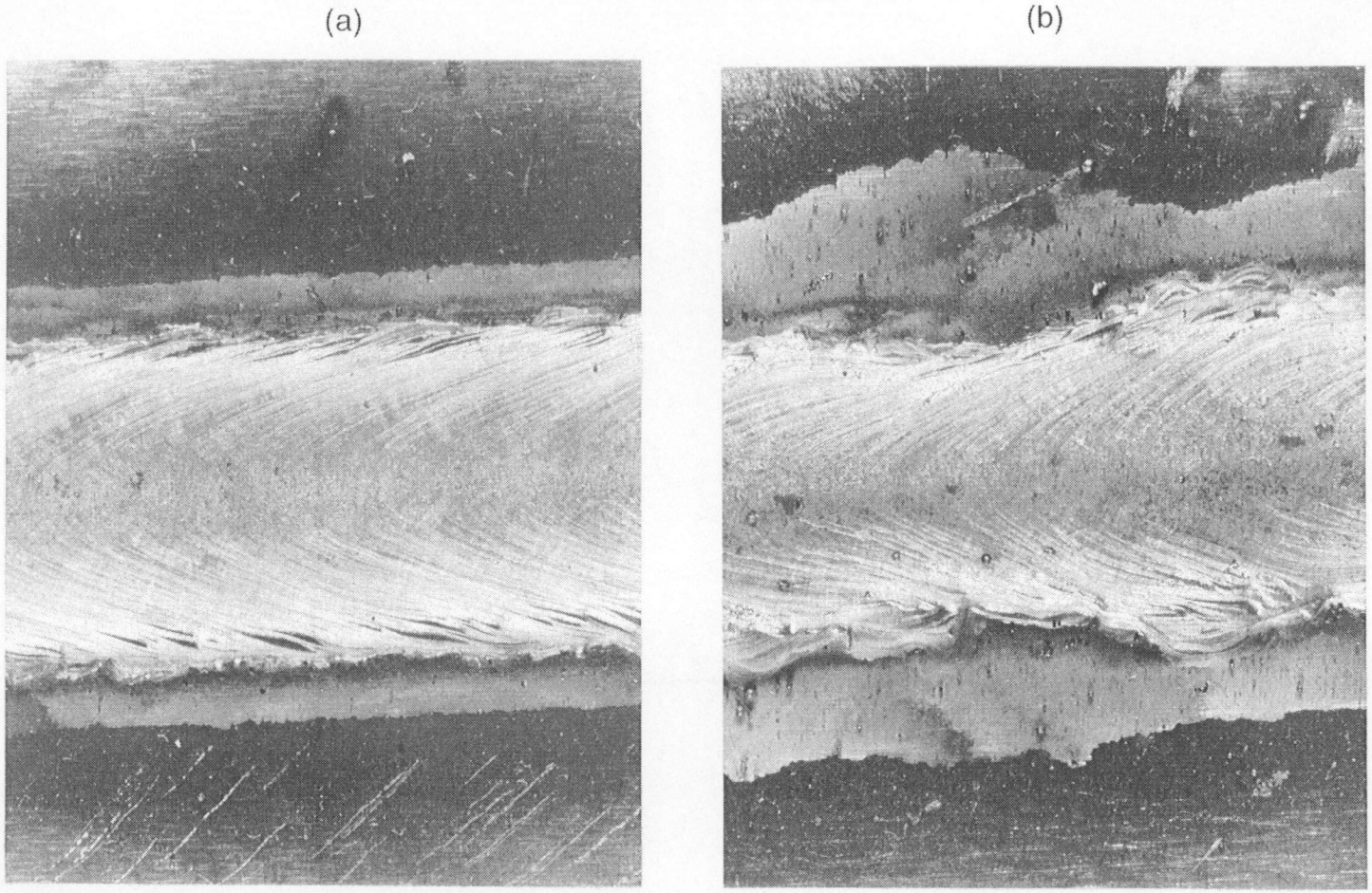


Figure 23. Surface appearance of GMA weld deposit on 304 stainless steel plate. (a) recommended argon-2%oxygen shield (b) argon shield

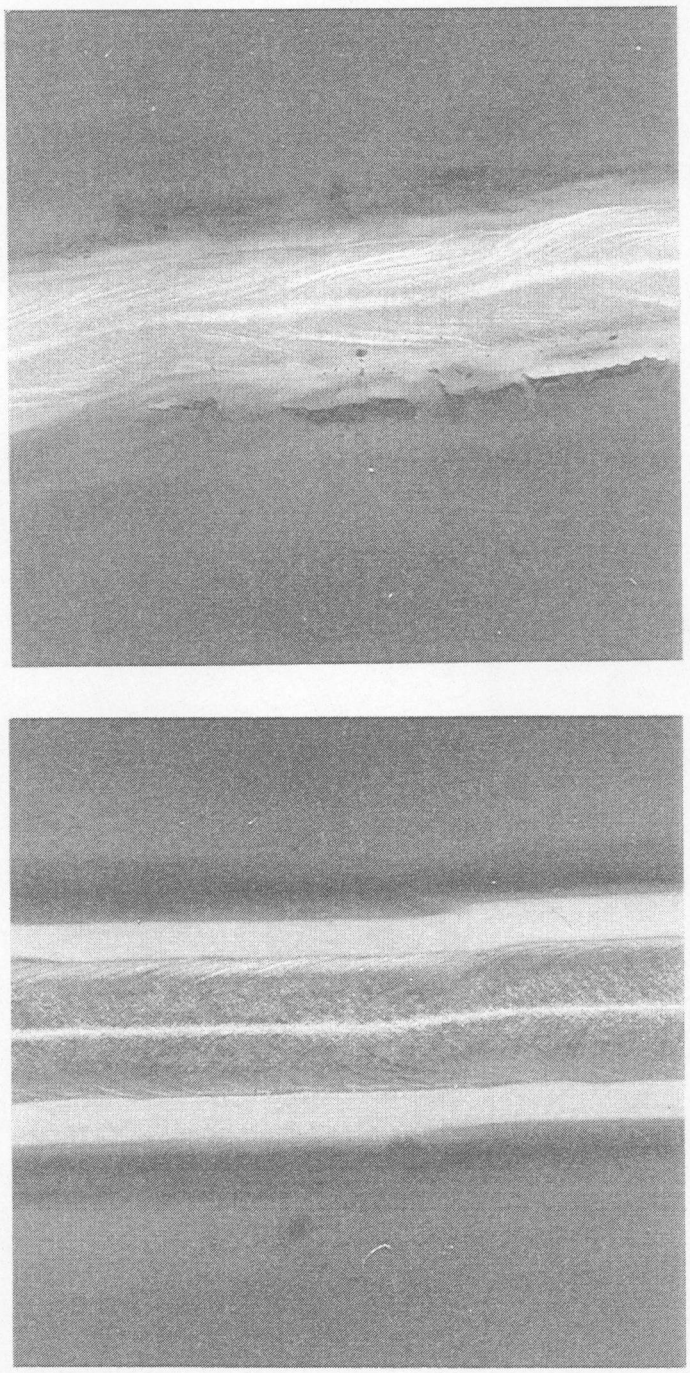
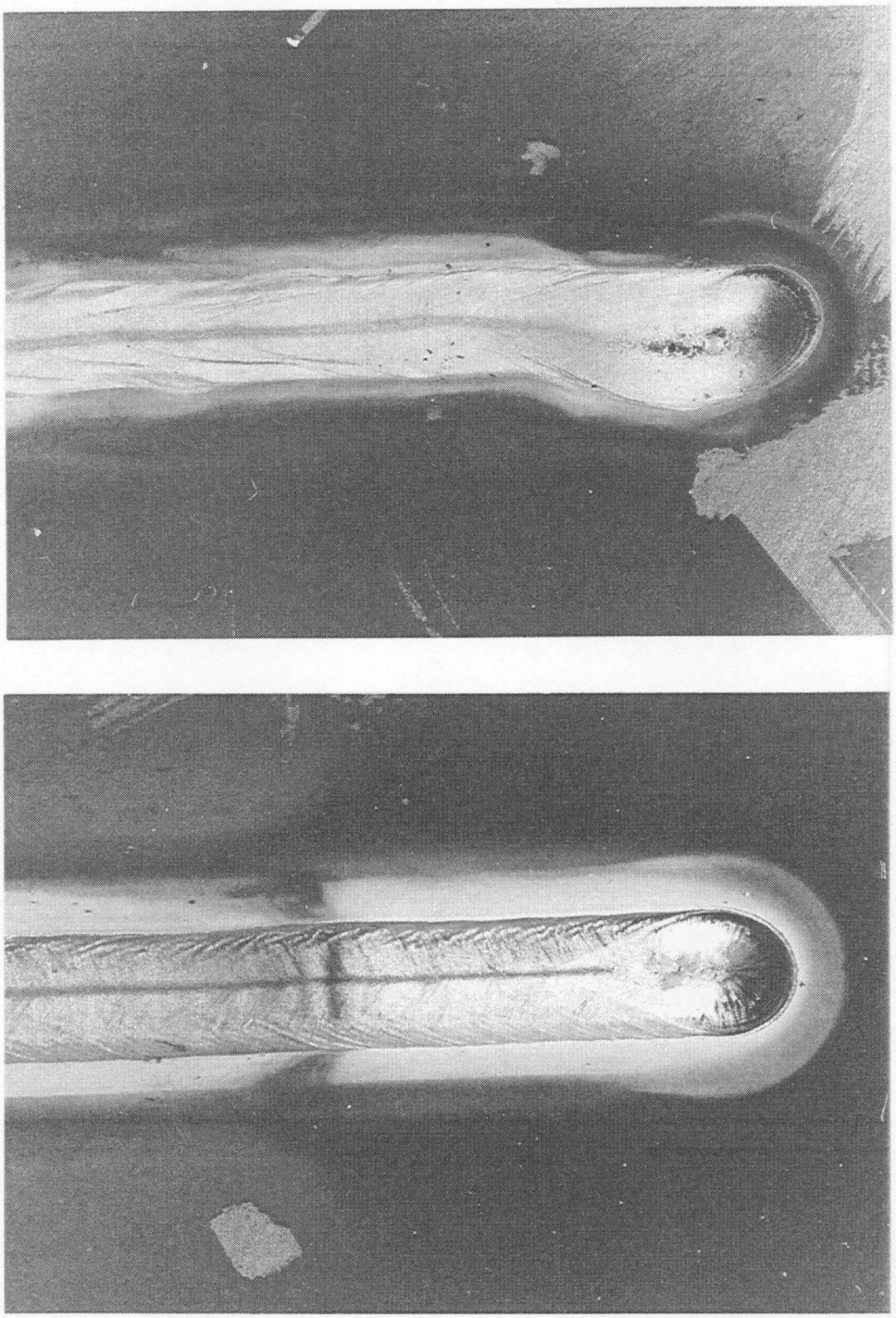


Figure 24. Effect of shield gas purity on surface appearance of autogenous 316 GTA weld. (a) argon shield contaminated with approximately 100 ppm oxygen and 300 ppm moisture (b) Nanochem purified argon shield

as indicated by the wavy weld bead. No defined gas coverage zone could be detected as the region adjacent to the weld was covered with soot.

In comparison, the sample welded with the same contaminated gas which was Nanochem-purified before use was clearly superior (figure 24b). The weld was extremely straight, thus indicating a stable arc, unlike the wander evidenced in the sample welded with the contaminated gas. Also, the gas coverage zone on either side of the weld was sharply defined, thus indicating clean and effective shielding. The large gas coverage zone also suggests that a higher welding speed can be used when welding, without compromising the shielding effectiveness. This feature clearly offers an added superiority over welds made with contaminated gas.

The differences discussed above are further reinforced by figures 25a and 25b, which represent the weld craters of the same welds described above made with contaminated gas and purified gas, respectively. The oxidation of the contaminated gas weld is clearly attested by its dark appearance in comparison to the shiny weld-stop/crater of the purified gas weld. The surface at the exact weld stop (figure 25b) in the sample welded with purified gas also appears shiny in comparison to the rest of the weld, which appears darker. The crater area, where the arc is finally extinguished, benefits from continuous shielding during the post-purge cycle. In contrast, the rest of the weld is subject to oxidation immediately after the welding torch has progressed away from it. Hence, the effects of shield gas contamination are most easily visible in the crater area, uninfluenced by atmospheric oxidation.



(a)

(b)

Figure 25. Weld crater of GTA autogenously welded 316 stainless steel.
(a) Argon shield contaminated with oxygen and moisture
(b) Nanochem purified argon shield

6.3.2. **316 Stainless Steel Polarization Results**

The polarization scans of the as-welded surfaces tested in 6% ferric chloride solution are indicated in figure 26; the shapes of the scans are similar. However, the overall anodic current density for the sample welded with Nanochem purification is less than that welded with contaminated gas. The difference in anodic current density is very high. A difference of 8000 uA/sq.cm in the anodic current density (at the reverse potential) was observed in the two samples. Apparently, the corrosion behavior of the regular welded austenitics is significantly influenced by shield gas purity.

However, it is interesting to note that the free corroding potential of the sample welded with contaminated gas is more noble than the sample welded with purified gas (170 mV as opposed to 75 mV SHE). This difference is expected because the sample welded with contaminated gas has inherently more oxide on its surface caused by the shielding gas impurities. This tenacious oxide film is probably responsible for the ennoblement observed. This feature, however, does not indicate any superiority because both 316 weld samples were found to corrode actively. Corrosion has occurred as indicated by the hysterisis loop generated upon reversing the potential. (The loop is generated when the current, upon reversal of potential, does not trace back along the initial up-scan curve.) This occurs because the current density on the down scan is higher than that observed for every corresponding potential on the up scan. Examination of the as-welded surfaces after corrosion testing revealed no pits, thus indicating uniform corrosion.

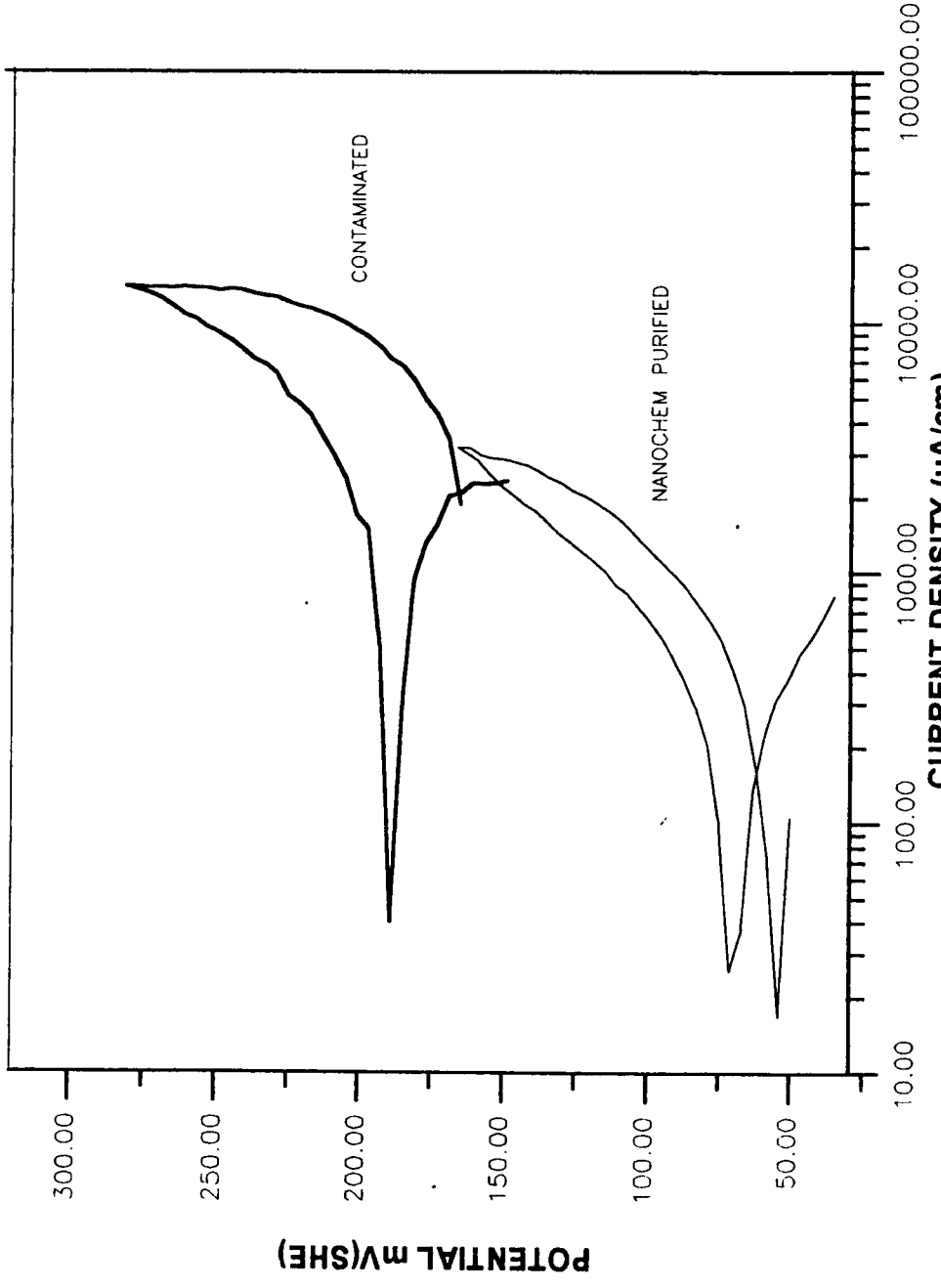


Figure 26. Cyclic polarization test results of GTA welded 316 stainless steel alloy welded with and without purification of contaminated argon shielding gas

6.4. **Titanium: Autogenous GTA Weld Tests**

In the test welds made using the GTA process, the shielding gas was contaminated with approximately 100 ppm oxygen and 300 ppm moisture moisture for two test welds. The same gas was then passed through the Nanochem purifier and a weld was made with the repurified gas at the same welding parameters as was employed for welding with the contaminated gas. One weld was then made with the as-bottled helium gas.

The most distinct characteristic observed was the difference in weld bead colors made with and without purification of the contaminated gas. The contaminated gas welds were strikingly blue in appearance throughout the weld zone, especially in the weld crater, indicating that impurities in the gas were responsible for the colored oxide film formation (figure 27a). The samples welded with purification, however, were especially shiny white in the weld stop (figure 27b). This difference attests to the efficiency of the Nanochem unit in filtering the moisture impurity added to the shield stream. In the sample welded using the bottled helium, the weld stop was as shiny as the sample welded with the Nanochem-purified gas. This fact indicates that the bottled helium gas was of sufficiently high purity.

The samples made with purified gas exhibited very slight "blueing" along the length of the weld. This slight oxidation observed is primarily due to the lack of shielding after progression of the weld torch away from the hot fusion zone. The use of a trailing shield can possibly eliminate such blueing. The fact that the weld crater area, exposed to the shielding gas until the weld had completely cooled (25 seconds), was blue only in the sample welded with contaminated

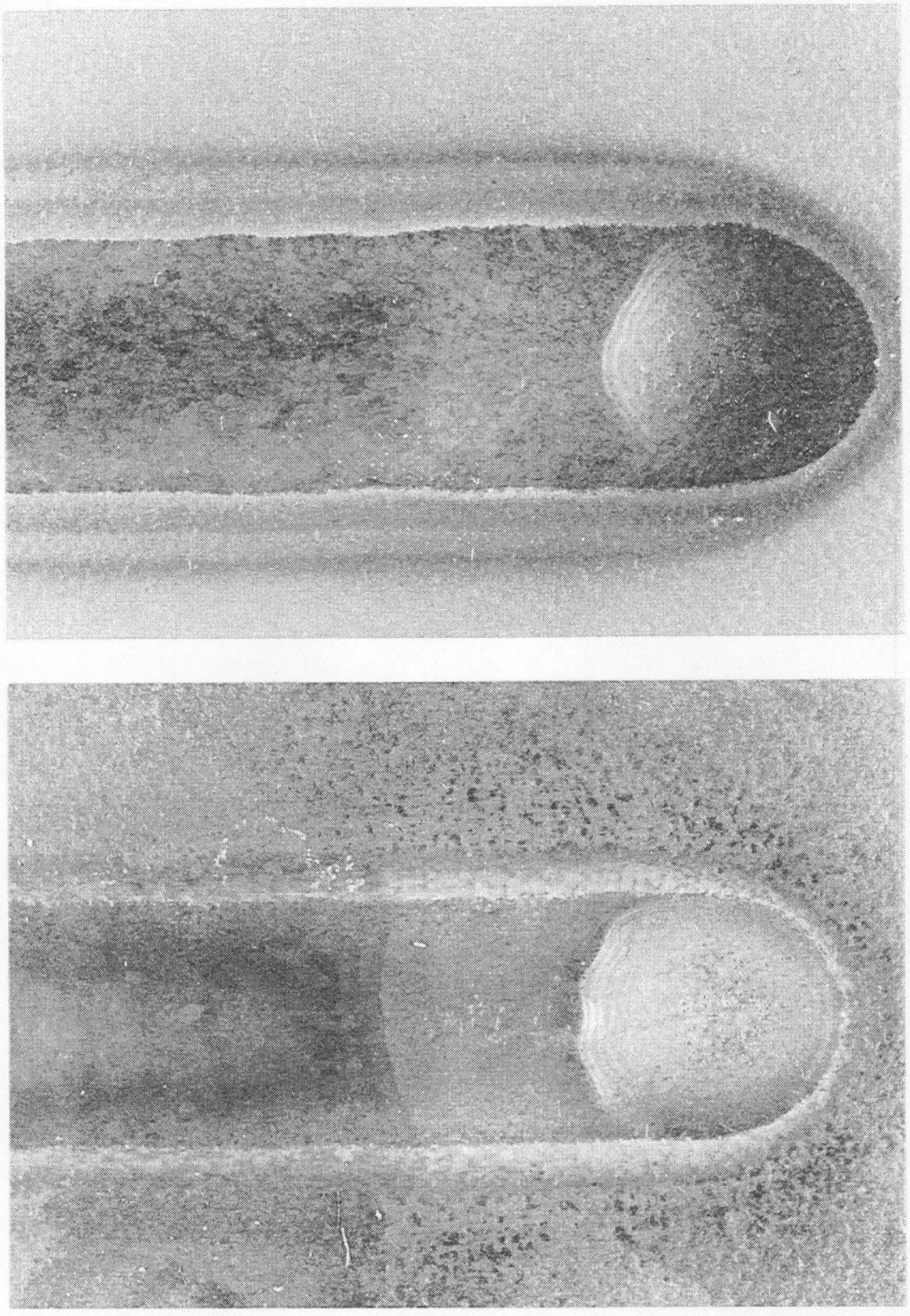


Figure 27. Effect of shield gas purification on the surface appearance of GTA autogenously welded titanium. (a) helium shield contaminated with 300 ppm moisture (b) Nanochem purified helium shield, originally contaminated with 300 ppm moisture

gas and shiny white in the sample welded with purification, is solely attributed to the purity of the shielding gas. This finding is in agreement with popular evidence that oxidation colors are characteristic of titanium welds shielded with contaminated gas. Beyond this difference, however, no other difference was detected. Both samples were bend-tested and indicated no difference in ductility.

Given the fact that the amount of moisture added to the shield gas was quite considerable for a material such as titanium, known for its ease of oxidation, the weld made with the contaminated gas was not grossly degraded in appearance as might have been expected. This result is attributed to three reasons. First, moisture must have been partly adsorbed onto the walls of the gas line as a result of which not all the calculated 300 ppm moisture must have been carried into the weld pool. Second, the mositure dissocation in the weld arc resulted in the release of hydrogen and oxygen in the shield above the weld. Consequently, the reducing effects of hydrogen must have offset the oxidizing effects of oxygen. The two factors acting in concert were probably responsible for relatively mild effects manifested in the blueing of the weld fusion zone. In any event, literature indicates that welds in titanium with hydrogen contents near 150 ppm or less can still be made without deleterious effects (porosity). Literature also indicates that even bubbling the shielding gas through water (as in this experiment) does not affect the porosity level of the welds [63].

As far as the blue colorations observed are concerned, most fabricators use mechanical or chemical cleaning methods to clean the welds if required for the end use of the product. No problems are posed by such surface films.

However, use of purified gas offers the possibility of eliminating the need for such post-weld operations. In any case, the color of the completed weld however, does indicate the effectivenss of shielding used. A bright , silver metallic luster, as seen in the experiments conducted with purified gas, indicates good shielding practice and gas purity.

6.5. **Aluminum 1100: Autogenous GTA Weld Tests**

The effects of shield gas contamination on GTA autogenously welded 1100 aluminum were clearly discernible immediately after the welding. The weld made with contaminated gas was characterized by a sooty-black, gas coverage zone, irregular weld progression, and fusion-zone oxide cracking (figure 28a). The surface of the weld zone was also dull. In comparison, when welded with Nanochem-purified gas, the weld was straight and exceedingly shiny (figure 28b). Interestingly, the shiny surface of the purified gas sample actually appears black (only in the photograph) in comparison to the surface of the sample made with contaminated gas. Both pictures, however, were taken at the same magnification and at the same level of subject illumination. Clearly, this black photo appearance suggests that the light reflectance level of the surface welded with Nanochem purification is so high (in comparison to the surface of the sample welded with contaminated gas), that at the same camera settings, it actually over-exposes the film. This finding holds potential in that developing a new procedure based on reflectivity readings of weld surfaces may be a feasible method of quantifying the effect of shield gas impurities on weld surface degradation.



Figure 28. Effect of shield gas purification on the surface appearance of GTA autogenously welded 1100 aluminum. (a) Moisture contaminated helium shield (b) Nanochem purified helium shield originally contaminated with moisture

The coupon welded with regular bottled helium indicated a surface appearance almost exactly similar to the sample welded with Nanochem repurified helium. This finding suggests that the bottled gas purity was high enough and even comparable to the purity of the gas obtained after Nanochem purification. The high bottled gas purity, however, should not be regarded as a norm. In field situations the inconsistency in bottled gas purity is a well known fact.

6.5.1. **Aluminum 1100: Corrosion Testing**

The two aluminum 1100 coupons welded with contaminated gas and with repurified helium gas were corrosion tested using electrochemical anodic polarization. The test results, depicted in figure 29, indicated that the 3% NaCl solution used (sea-water type medium) is extremely aggressive and causes active corrosion irrespective of the surface condition of the aluminum weld surface.

For the autogenously welded 1100 aluminum, the sample welded with purification exhibits an anodic current density of 1000 uA/sq.cm at the reversing potential. In comparison, the sample welded without purification exhibits an anodic current density of 7000 uA/sq.cm at the reversing potential. This difference, although suggesting a beneficial effect, is not really significant given the active corrosion experienced by both samples.

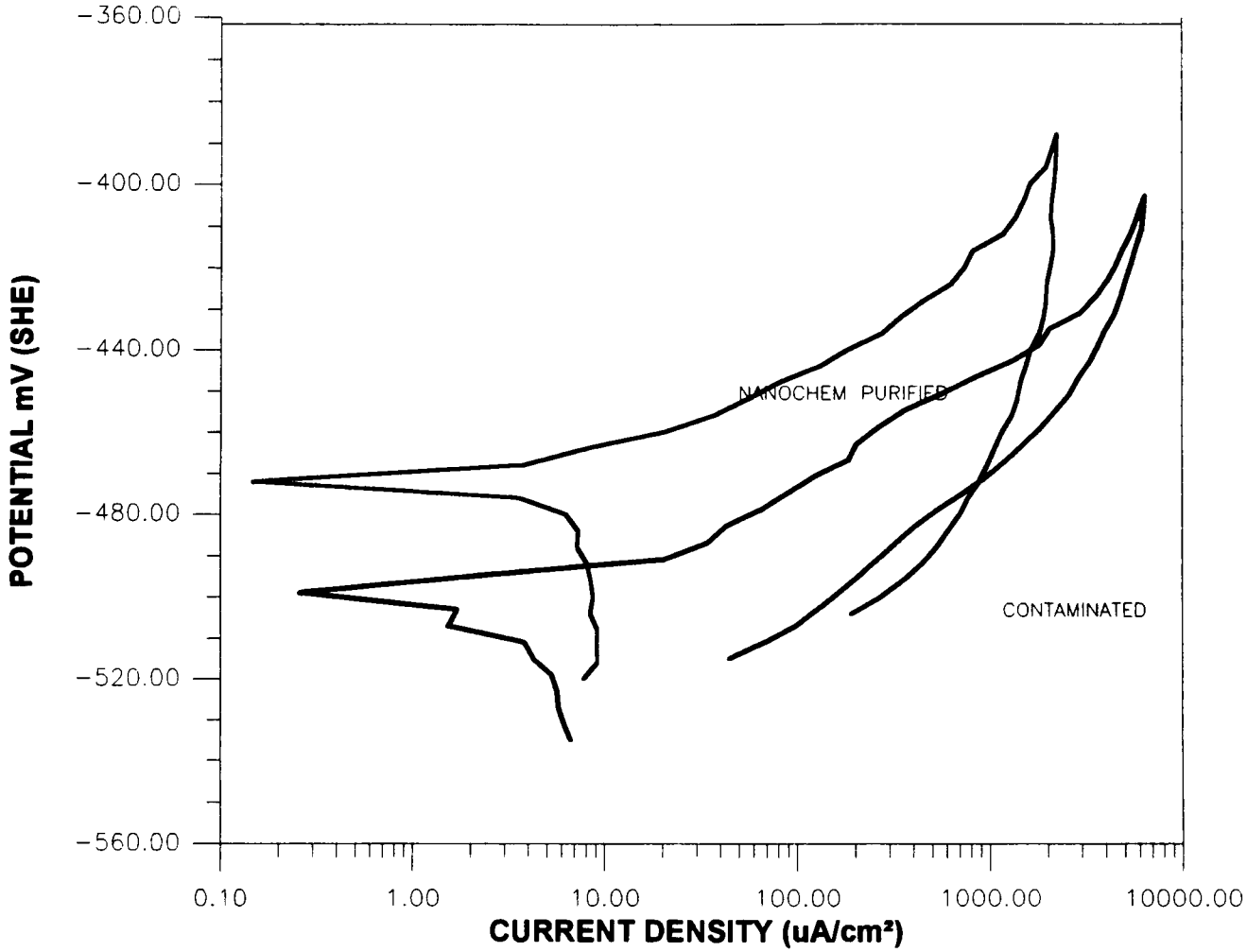


Figure 29. Anodic polarization scans of 1100 aluminum welds in 3.5% NaCl solution, shielded with and without purification of contaminated helium gas.

6.6. **1100 GMA Weld Tests**

6.6.1. **Surface Appearance**

As emphasized in previous sections, the as-welded surface appearance of a weld is a strong indicator of the overall integrity of a weld. Inferior weld appearance often warrants rejection at the fabrication stage itself. Similar to the behavior of other alloys tested in the program, the GMA welds made on the 1100 aluminum indicated a strong distinction between the purified and contaminated gas welds. These representative differences are pictorially depicted in the attached figures. Figures 30a and 30b represent a typical comparison between a "pure gas" weld and an "impure gas" weld. The black soot, rough surfaces, and the few weld and oxide cracks seen on the sample depicted in figure 30b were typical of all samples welded with the oxygen-and-moisture contaminated argon shielding gas.

The gas coverage zone area excluding the fusion zone was characteristically deposited with a sooty, non-adhering deposit. The samples welded with the same contaminated shielding gas, which was repurifed before use with the Nanochem system were, by contrast, clean and shiny. Sooty deposit, when present, was confined to regions out of the range of the gas shielded zone. The observations decribed above are presented pictorially in figure 30 and subsequent figures. Because the samples had been scrupulously cleaned prior to welding, the sooty deposit was unlikely to have come from any plate residues. (The plates had been rinsed with acetone and scrubbed with stainless steel wool until the superficial layer was completely removed,



(a)

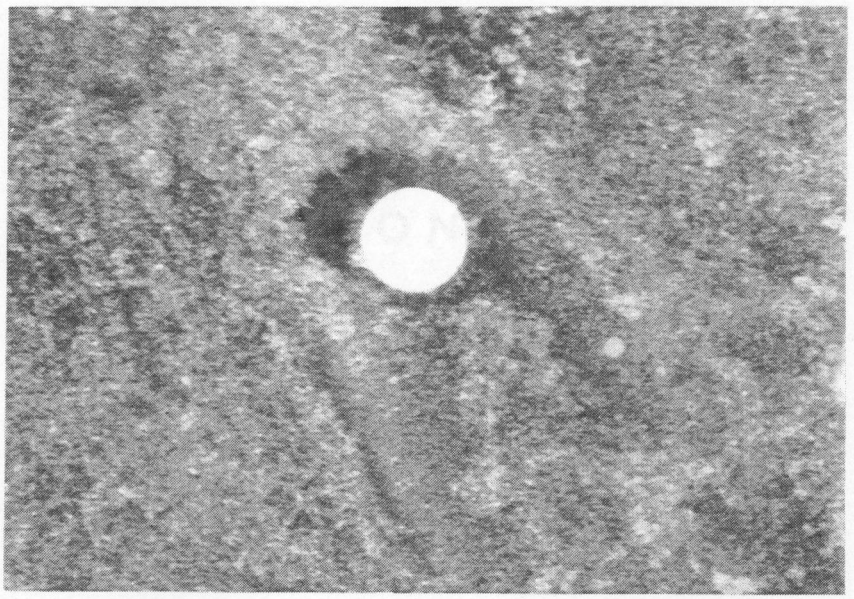
(b)

Figure 30. Effect of shield gas purification on the surface appearance of GMA welded 1100 aluminum. (a) Nanochem-purified argon shield originally contaminated with oxygen and moisture (b) argon shield contaminated with approx. 100 ppm oxygen and 300 ppm moisture

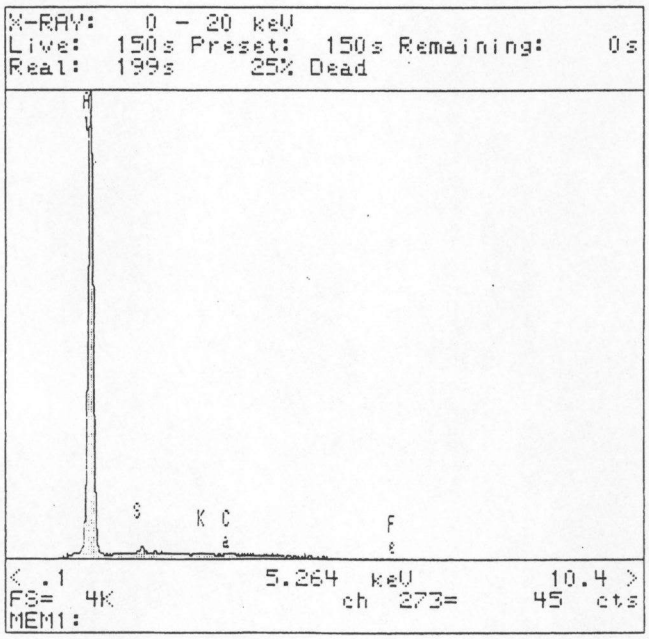
exposing a fresh, oxide- and-dirt free surface, and then rinsed in acetone again.) It was necessary to investigate the soot composition in order to determine its source. Accordingly, SEM studies in conjunction with EDS analysis were conducted on the soot taken from a representative sample. The carbon tape "mechanical imprinting and extraction" procedure used was successful in transferring a perfect replica of the soot from the specimen surface to the SEM specimen holder. SEM and EDS studies established that the soot is essentially condensed aluminum of submicron size, as attested by the high magnification photograph in figure 31a and the EDS spectrum in figure 31b. The aluminum soot (black area) is so fine that its morphology cannot be resolved even at 2 microns. However, a larger particle of condensed aluminum can be seen (white sphere), which further testifies that the soot is composed of fine, condensed aluminum particles.

An explanation as to the reason why welds made with contaminated gas contained more soot in the gas coverage zone than welds made with purified gas is couched in the theory that the fine condensing particles are severely oxidized and thus form a non-coherent, non-adhering mass of sooty oxide. In any event, the effect of soot is only superficial and is easily erased by power brushing (figures 32 a&b).

Surprisingly, even with the presence of moisture as high as 240 ppm in the shield gas, little porosity was apparent in the weld. The near absence of gross surface porosity may possibly be attributed to adsorption of moisture on the surfaces of the gas plumbing. Such adsorption prevents the injected moisture from reaching the weld pool. However, in these experiments, such adsorption phenomena was accounted for by employing a purging time of over



(a)



(b)

Figure 31. Soot analysis of 1100 aluminum alloy GMA welds. (a) SEM examination (b) EDS spectrum of weld soot.



Figure 32. Effect of power brushing on soot removal in GMA welds of 1100 aluminum alloy. (a) Nanochem purified argon shield (b) contaminated shield

one hour. This procedure allowed the gas delivery lines to get fully saturated with moisture, thus allowing the entire injected moisture to reach the weld pool without being adsorbed on the gas lines.

A different explanation is also offered for explaining the absence of surface porosity and is couched in the literature describing work performed by other researchers [39]. Data in the literature indicates that when welding aluminum, oxygen in the shielding gas up to an extent of 5% can be beneficial in terms of preventing the formation of porosity. Because the shield gas in this work also contained 94 ppm oxygen, it is fitting to consider this aspect also when explaining the absence of gross weld porosity.

The effects of gross contamination (240 ppm moisture and 94 ppm oxygen) in 1100 welded aluminum are shown in figure 33. Severe hot cracking and extensive surface oxide formation are apparent. Figure 34 depicts (in black and white), the typical differences effected by purification of the shield gas using Nanochem. The typical features of the contaminated and purified gas welds were not altered by changing the contamination levels (20 to 100 ppm oxygen, and 40 to over 300 ppm moisture). The differences effected by purification were most apparent in the weld craters, as depicted in figure 35.

Bend testing of the aluminum samples indicated equal ductilities in samples welded with and without gas purification. This behavior is understandable given the inherent ductility of 1100 aluminum.

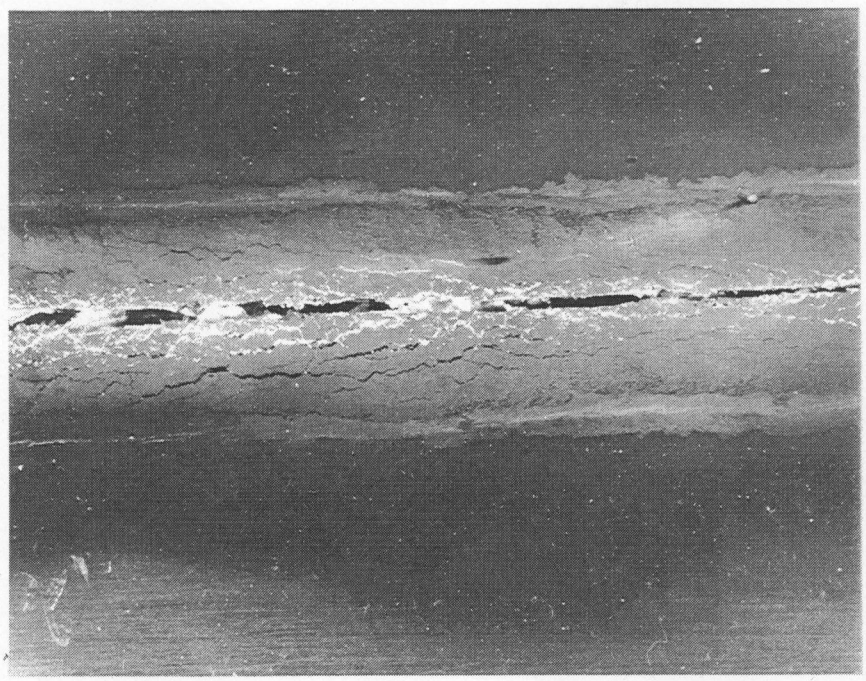


Figure 33. Effect of high contaminant level (158 ppm oxygen, 375 ppm moisture) in the shielding gas on weld appearance of 1100 aluminum. Weld cracks and oxide cracks visible.

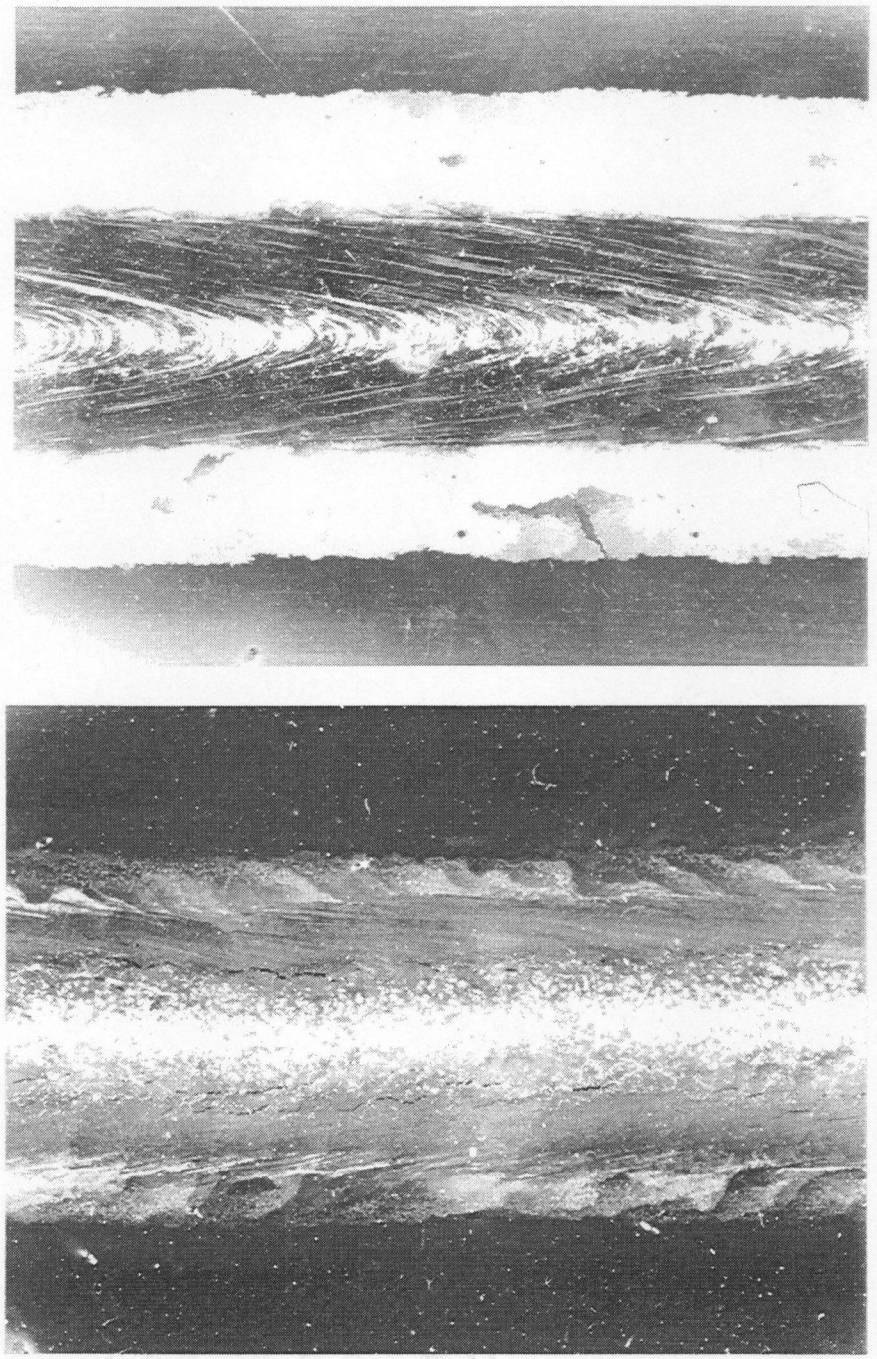
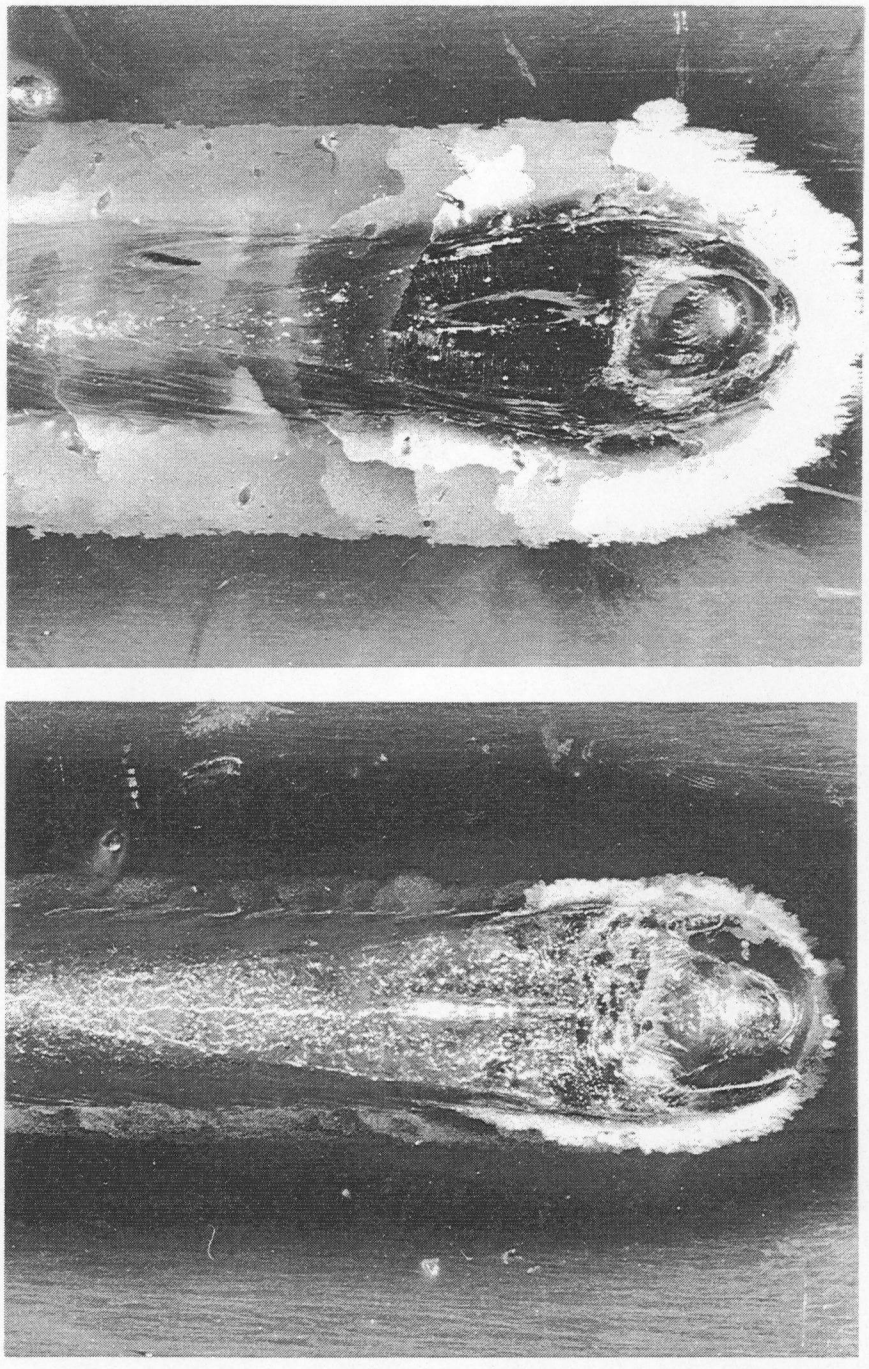


Figure 34. Photograph highlighting the difference between 1100 GMA aluminum welds shielded with (a) Nanochem purified argon gas and (b) moisture and oxygen contaminated argon gas.



(a)

(b)

Figure 35. Effect of shield gas purification on the appearance of 1100 aluminum GMA weld craters. (a) Nanochem purified shield (b) contaminated gas shield (94 ppm oxygen and 88 ppm moisture)

6.6.2. **Arc Sound Studies**

A perceivable auditory difference was noticed in the arc when welding with contaminated and repurified shield gas. Even when using the same welding parameters, there was a difference in metal transfer modes, attributable only to the purity of the shield gas. A slight degree of spatter was associated with the transfer mode when welding with the purified gas. This observation was attested by the audible sound emanating from the arc, which was unlike the sound associated with the spray transfer mode so clearly observed when welding with contaminated gas.

The arc sound was studied in a more refined fashion using graphical representation of the recorded arc sound in terms of frequency and noise level. The attached computer processed sound data show a definite difference. The graphs represent a variation of frequency and decibel level over a four second duration. Figure 36a indicates the variation of frequency and arc noise when the welding was carried out using contaminated gas repurified using Nanochem. The contaminated gas originally contained 240 ppm moisture and 94 ppm oxygen. The frequency and decibel levels are characterized by definable extreme peaking and cresting, indicating a noisy arc.

In contrast, the block of sound data for a weld made with the same contaminated gas (19 ppm moisture and 8 ppm oxygen) without purification is depicted in figure 36b. The frequency graph is almost a flat, solid band, and the characteristic peaks are hardly discerned owing to their proximity. This characteristically indicates a consistent sound level and therefore a consistent level of metal transfer, such as "spray". These evident differences in arc sound

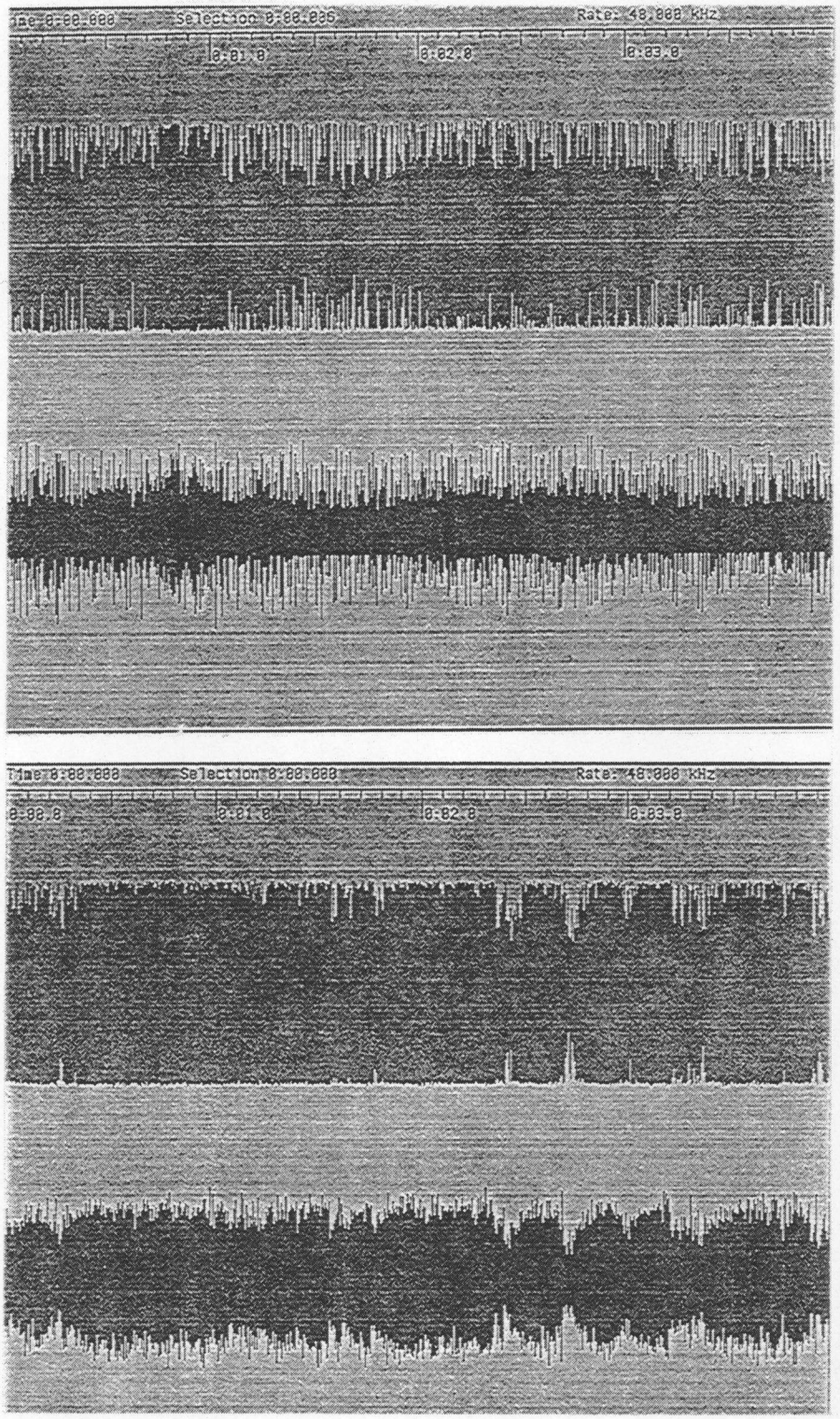


Figure 36. Difference in arc sound levels in terms of decibel level and frequency in 1100 GMA aluminum welds shielded with (a) Nanochem purified gas and (b) contaminated gas.

quality arising from shield gas purity imply that, all other parameters being equal, metal transfer in GMA aluminum welds is seemingly influenced by the level of contaminants in the shield gas. The difference stems from the fact that the ionization potential of the shield gas is altered by the presence of moisture and oxygen. This alteration is then manifested in change in the voltage and arc current characteristics. However, the difference is not significant in terms of affecting weld quality and properties.

6.7. **Aluminum 5086: GMA Weld Tests**

The effect of contaminants was evident immediately after welding. A stark contrast was observed in the appearance of the as-welded coupons, welded with and without purification. In fact, even during the actual welding, the arc sound seemed to suggest that the level of contaminants in the shield gas also influences the metal transfer modes. In experiments with GMA welded 1100 aluminum, arc sound data was studied to examine any differences in the transfer mode. The arc sound characteristics when welding with the 5556 wire were the same as those when welding with the 1100 wire in the earlier experiments. For the same welding parameters, when welding using Nanochem, the metal transfer mode was more noisy. The noise is suggestive of a more globular type of metal transfer. However, the metal transfer mode is not a significant aspect in terms of affecting the final weld properties. The only factor that may be affected by the mode of transfer is the deposition rate and the welding position.

6.7.1. **Helium Shielded Weld Coupons**

Figure 37a shows the appearance of a typical weld bead made with helium shielding gas that was contaminated with approximately 300 ppm moisture. In contrast, figure 37b shows a weld made with the same contaminated gas, which was repurified with Nanochem prior to reaching the torch. The differences are readily visible. The surface of the weld shielded with Nanochem-purified helium is smooth and shiny. In contrast, the samples welded with the moisture-contaminated gas are characterized by black spots and a relatively rough and lusterless surface. On initial inspection of figure 37a, the black spots seem to be porosity defects. However, stereo examination confirms that they are black oxide colorations. Soot was also observed on both types of samples welded with and without gas purification. Soot of this nature was earlier determined to be condensed aluminum of sub-micron size. It does not interfere with the properties of the welds. In multi-pass welding, such soot is simply wiped away before every pass.

6.7.2. **65Argon-35 Helium Shielded Weld Coupons**

Figure 37c shows the appearance of a typical weld bead made with helium-argon shielding gas that was contaminated with 300 ppm moisture and 100 ppm oxygen. Figure 37d depicts the appearance of the weld made with the same gas, but repurified with Nanochem before reaching the torch. The sample welded with Nanochem-purified gas clearly possesses a high luster.

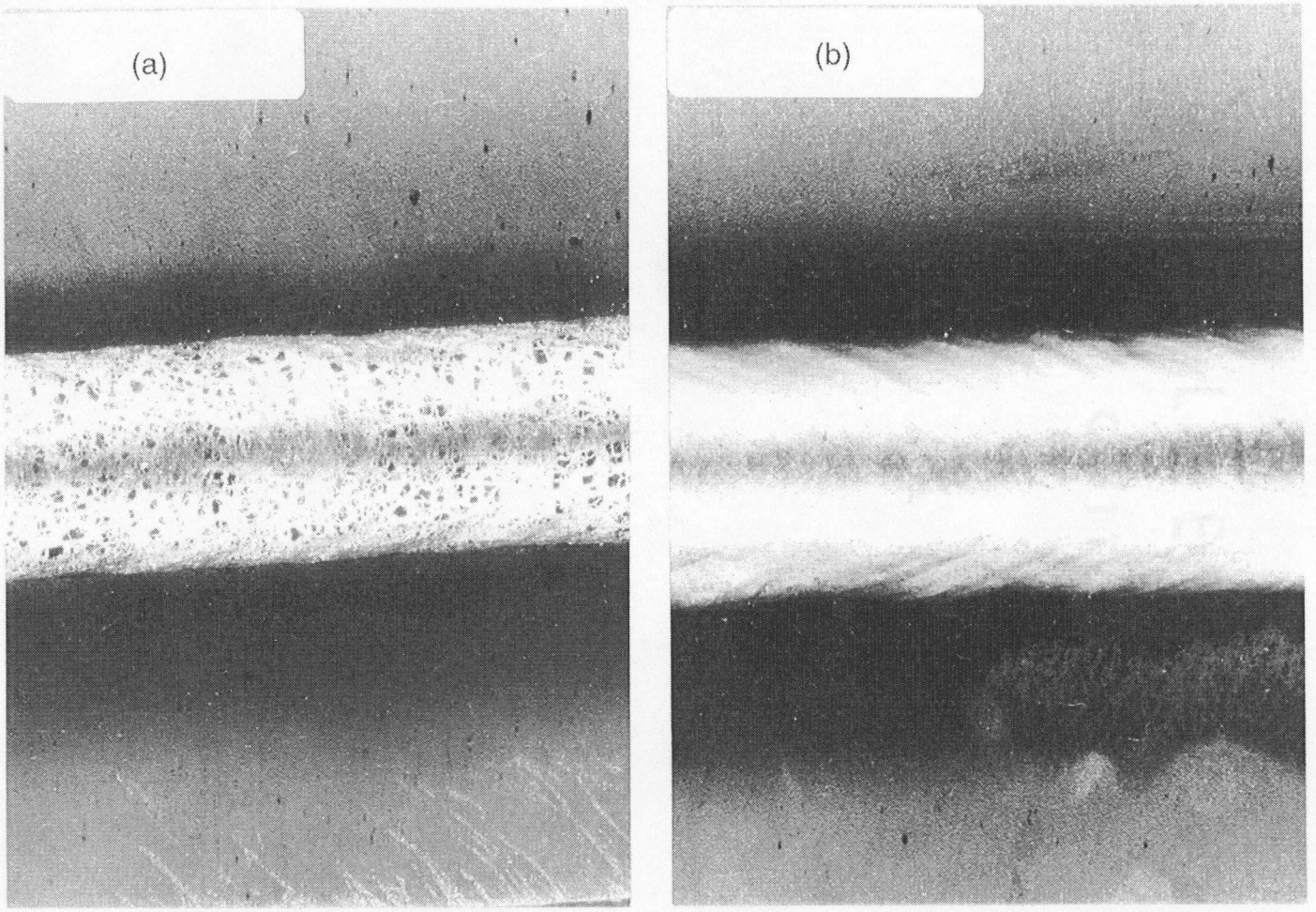


Figure 37. Effect of shield gas purification on the surface appearance of 5086 aluminum GMA welds. (a) Moisture-contaminated helium shield (b) Nanochem-purified helium shield

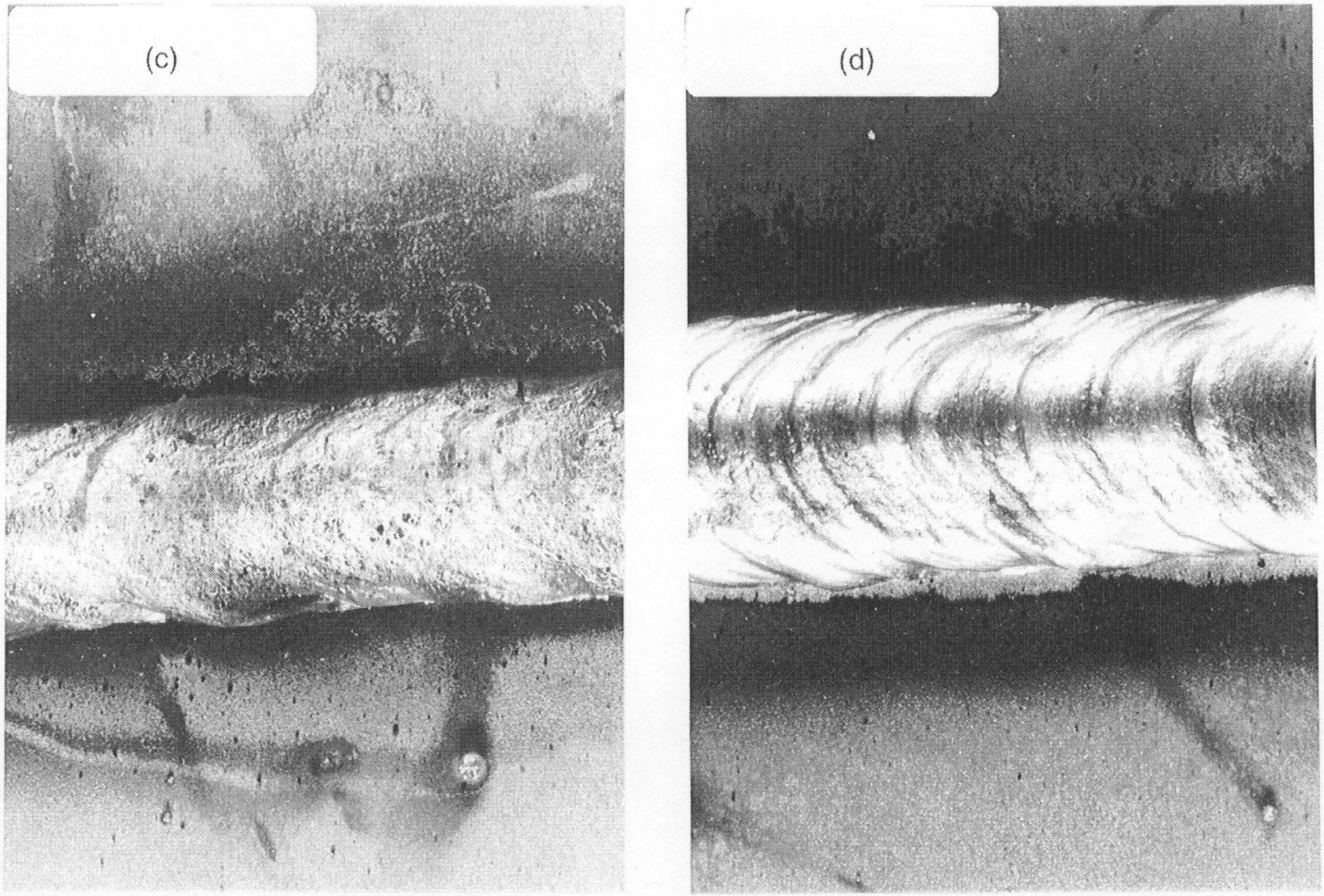


Figure 37. (continued) (c) moisture and oxygen contaminated 65Ar-35He shield (d) Nanochem purified 65Ar-35He shield

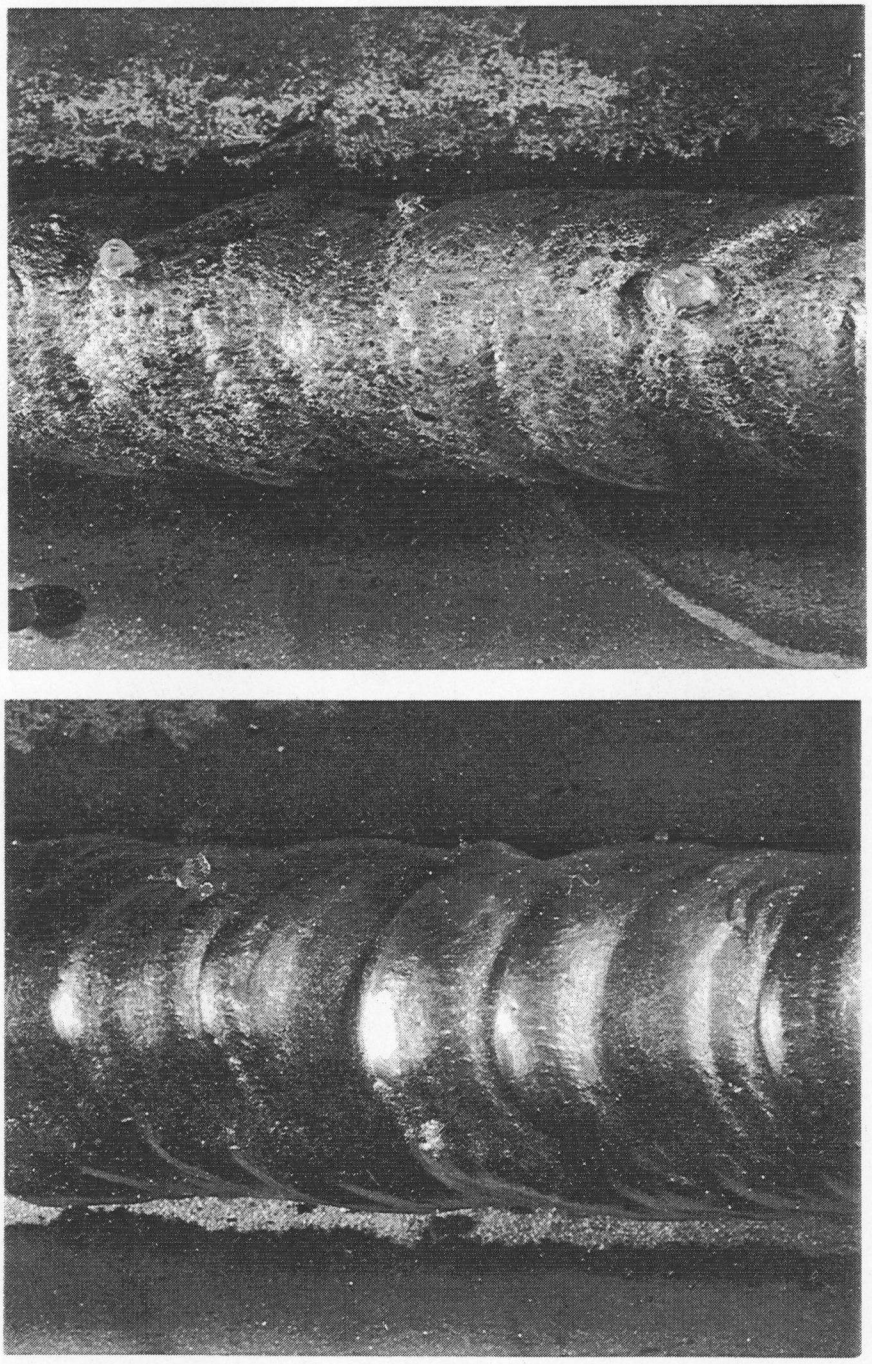
In contrast, the sample welded with the contaminated gas has a severely degraded appearance. As observed in figure 37c, the gas coverage zone adjacent to the weld is also unusual. Unlike the well-defined, black, aluminum oxide soot found in the other welded samples, the gas coverage zone is different in appearance. The zone is non-uniform with grey and white tones and suggests a non-uniform oxidation over the surface of the weld and fusion zone. Even during the actual welding, a lot of unusual activity was observed in the pyrotechnics of the weld. Tiny sparks were seen adjacent to the fusion zone during the course of welding. A close-up view of such a sample welded with contaminated gas is illustrated in figure 38a. The surface is characterized by rough spots and craters. As opposed to this unsightly appearance, a close-up of the Nanochem-purified weld indicates a weld bead surface that is extremely smooth and shiny (figure 38b).

6.7.3. **Bend Testing**

6.7.3.1. **Helium Shielded Coupons**

Bend testing was conducted for four representative coupons shielded with the following gases/gas conditions:

1. Contaminated 65Ar-35He. (100 ppm oxygen, 300 ppm moisture)
2. Same as 1, but repurified with Nanochem before use.
3. Contaminated He. (300 ppm moisture)
4. Same as 3, but repurified with Nanochem before use.



(a)

(b)

Figure 38. Close-up view of GMA welded aluminum 5086 surface showing the effect of shield gas purification. (a) moisture and oxygen contaminated 65Ar-35He shield (b)Nanochem purified 65Ar-35 He shield

Bend testing was carried out to examine the deleterious effect of contaminants in the shielding gas and the subsequent effect of Nanochem purification. The results of the bend tests are presented in figure 39.

The sample welded with Nanochem-repurified helium gas exhibited the most superior bend properties. A complete wrap around the bend-test plunger was achieved, without any indication of a single crack or defect. This superior behavior is in agreement with the relative appearance of the welds, since the coupons shielded with helium also appeared the best. In sharp contrast, the sample welded with the contaminated helium gas without purification exhibited poor bend ductility. The bend angle at failure was a mere 60 degrees. The ductility difference between the contaminated gas weld and purified gas weld in terms of bend angle was 100 degrees.

6.7.3.2. **65Argon-35 Helium Shielded Samples**

Both the coupons welded with the 65argon-35 helium mixture, however, failed during bend testing. A pictorial representation of the two bend-tested samples, welded with and without purification, is presented in figure 40. The coupons had been welded with moisture and oxygen contaminated argon-helium mixture. The failure of both samples welded with the 65Ar-35He shield mixtures emphasizes that although Nanochem gas purification safegaurds against weld degradation due to moisture and oxygen impurities, some purified mixtures may still be inherently inferior to other purified mixtures. Hence, in spite of purification, no improvement in mechanical properties was detected, unlike in the samples shielded with helium gas. Welding with super-pure gas

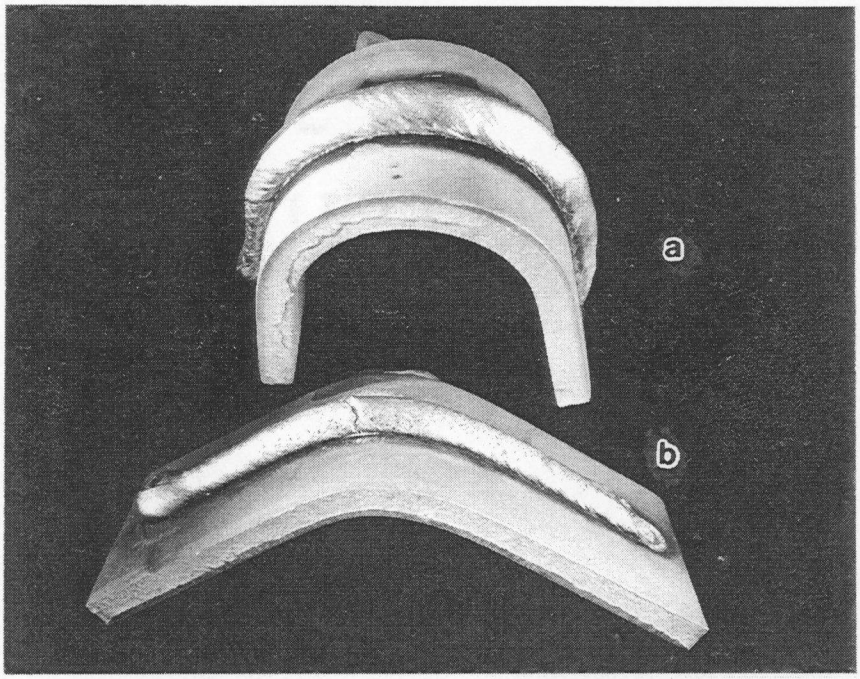


Figure 39. Appearance of 5086 aluminum GMA welds after bend testing until failure indicating difference in bend ductility. Sample A - shielded with Nanochem purified helium gas and sample B - shielded with moisture contaminated helium gas

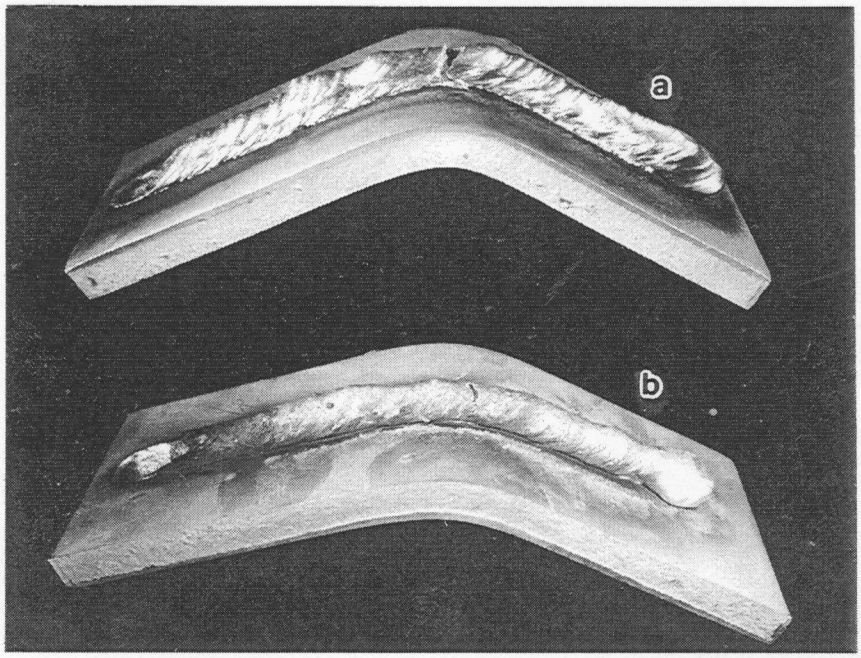


Figure 40. Appearance of GMA welded 5086 aluminum welds after bend testing indicating no difference in bend ductility. Sample A was shielded with Nanochem purified 65Ar-35He gas and sample B was shielded with moisture and oxygen contaminated 65Ar-35He gas

may therefore not necessarily guarantee improved bend ductility. The possibility of weld defects caused by reasons other than the shield gas impurities should always be considered when welding. Contamination may also arise from oils on the surface of the plate, moisture on the plate, atmospheric humidity, and strong wind drafts that cause momentary loss of shielding. In this set of experiments, the 65-35 argon-helium shield mixtures produced welds with similar bend properties irrespective of the gas purity level. The bend angles at failure in both samples was approximately 50 degrees. This value is low compared to the bend angle of 160 degrees exhibited by the sample shielded with the repurified helium gas.

6.7.4. **Metallography of 5086 Welded Aluminum Welds**

One representative coupon each was sectioned from the set of four welded aluminum coupons welded with different gases/gas conditions and prepared for routine metallographic examination. The most obvious effect of contamination was manifested as pores. Metallographic examination revealed a significant difference between the contaminated-gas and purified-gas welds for both types of shield gas mixtures used. Although pores were found in both types of samples (welded with and without purification), the level of porosity in the welds made with moisture-contaminated gas alone was visibly higher. The fact can be easily attested by figures 41a through 41d. Comparison of figures 41a and 41b, samples welded with contaminated helium gas and Nanochem-purified helium gas, respectively, clearly reveals a significant difference in the

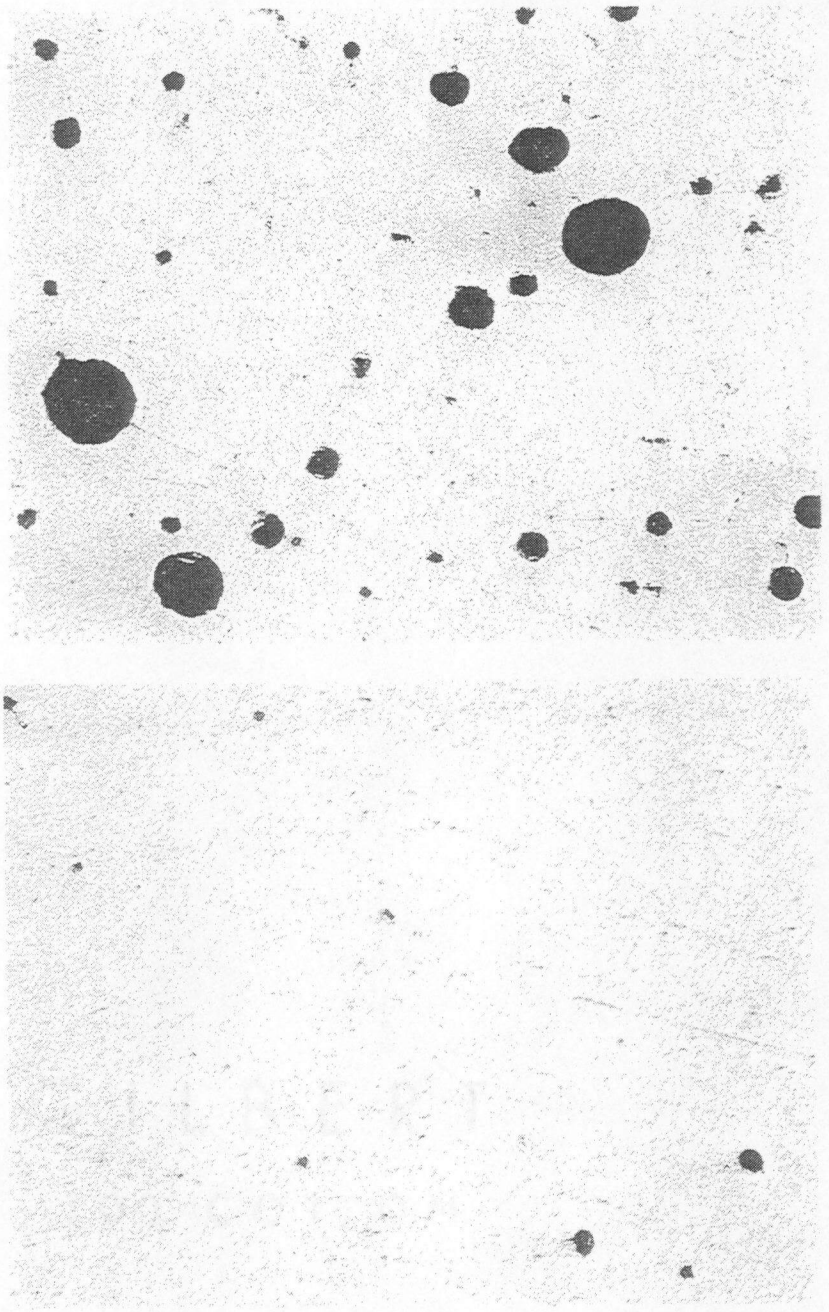
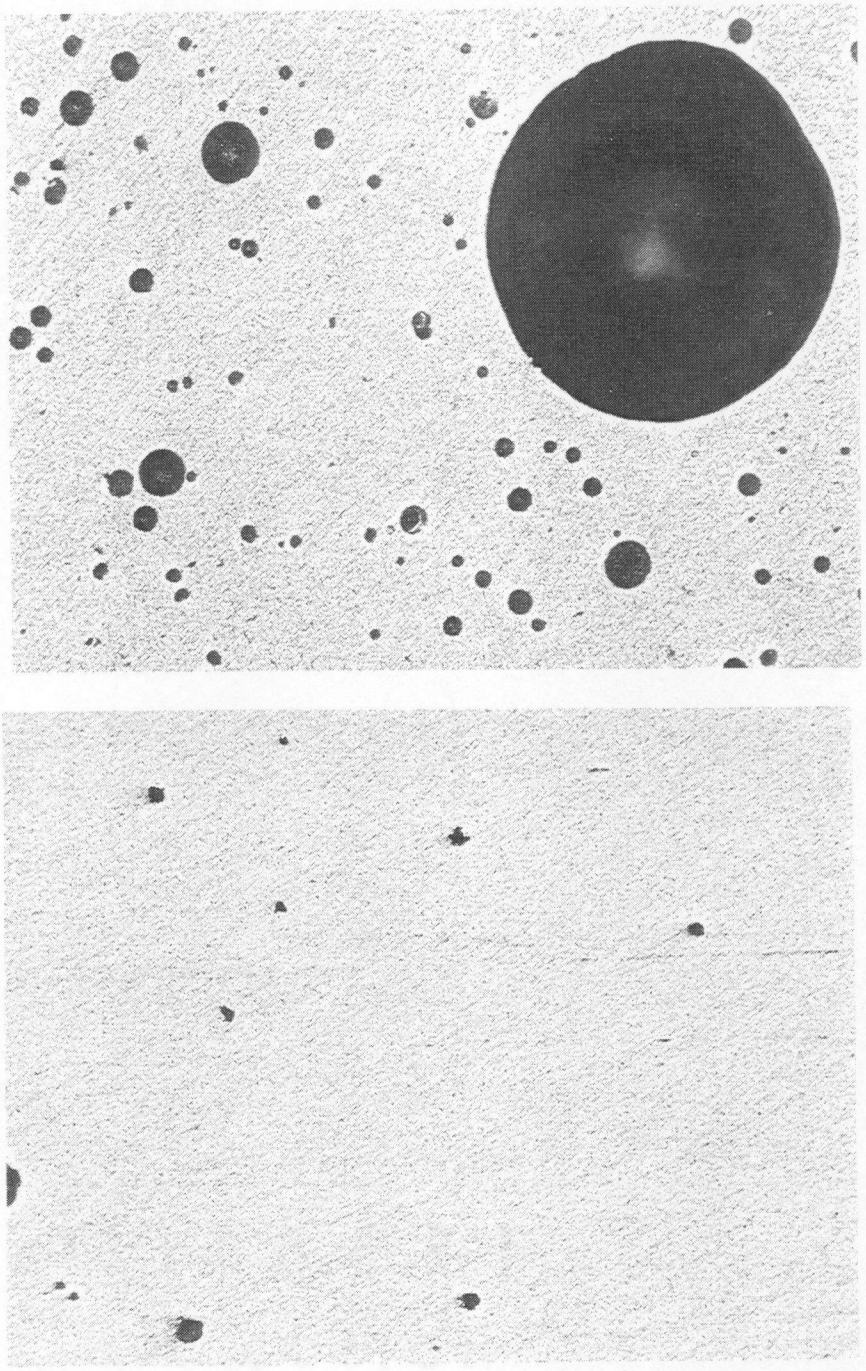


Figure 41. Metallographic section of GMA welded 5086 aluminum indicating porosity level as a function of shield gas purity.
(a) moisture contaminated helium shield (b) Nanochem purified helium shield



(c)

(d)

Figure 41 (continued). (c) moisture and oxygen contaminated 65Ar-35He shield (d) Nanochem purified 65Ar-35He shield

porosity levels. The occurrence of porosity is more pronounced in the samples welded without Nanochem purification.

Figure 41c indicates the typical porosity level in a section extracted randomly from one of the test coupons welded with oxygen- and moisture-contaminated 65argon-35helium shielding gas. Compared to the coupons shielded with Nanochem-purified gas (figure 41d), the degree of porosity observed is clearly higher in the weld shielded with contaminated gas.

6.7.5. Corrosion Testing

6.7.5.1. 3.5% Sodium Chloride Tests

The test solution used for testing the aluminum coupons was 3.5% wt. NaCl solution. The NaCl solution simulates sea water and hence the rationale for choosing it in the present study has been described in a previous section.

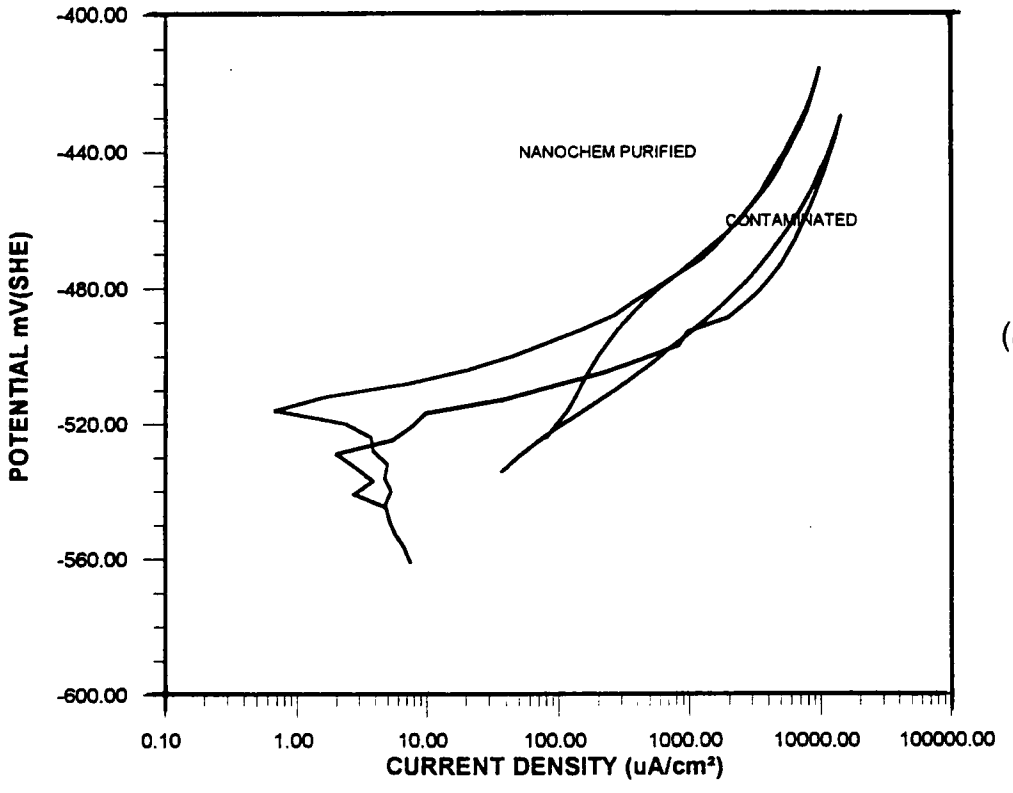
Owing to the significant differences in the appearance of the welds, large differences in corrosion were expected. The test welds made with the Nanochem-purified welds appeared shiny and clean. By contrast, the welds made with the contaminated gas without purification were poor in appearance. The welds made without any purification were characterized by heavy deposits of soot, a rough, lusterless appearance, and heavy oxides. Therefore, contaminated gas welds were expected to behave poorly in the corrosion tests. The electrochemical polarization results obtained with 5086 aluminum GMA welded coupons were similar to the results obtained with the 1100

autogenously welded aluminum. The polarization scans are depicted in figures 42a and 42b.

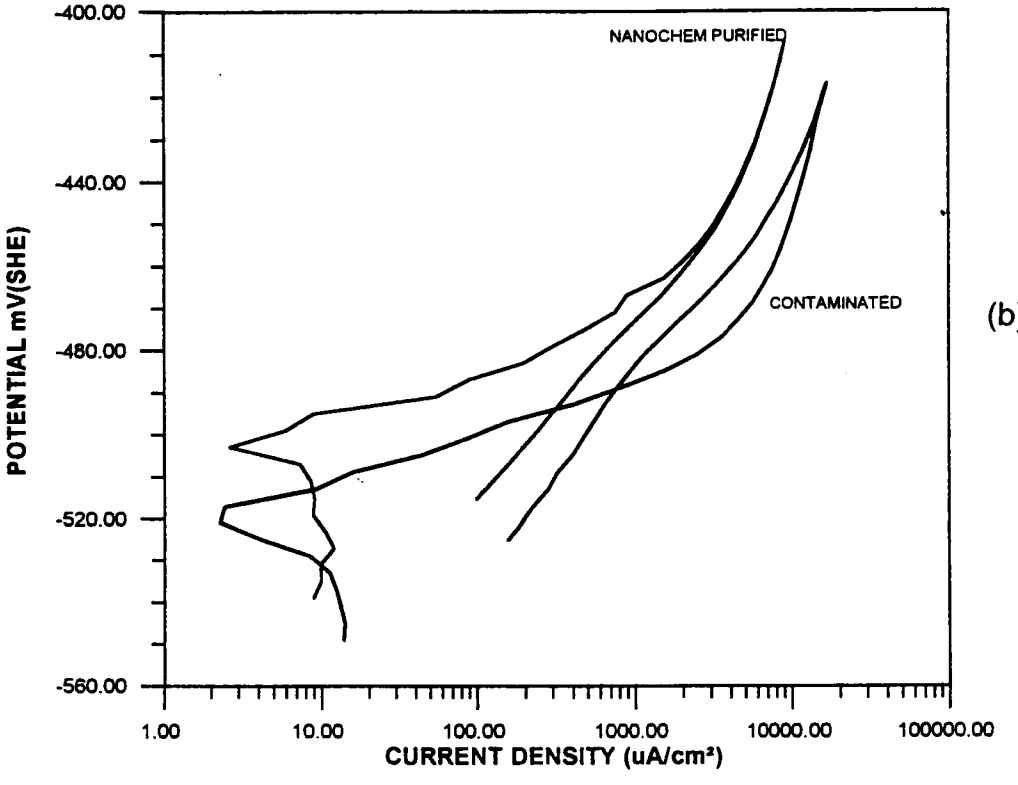
The general shape of the polarization scans is similar and indicates that all samples exhibited uniform corrosion. E_{corr} values for all samples ranged from -520 to -425 mV (SHE). For the samples welded without purification, anodic current densities at the reversing potential were approximately 10,000 uA/sq.cm. However, for the samples welded with purified gas, the corrosion current densities at the reversing potential were always less than 10,000 uA/sq.cm.

As seen in figure 42b, for the sample welded with purification using the 65 argon-35 helium shielding mix, the anodic current density at the reverse potential for the sample is 9000 uA/sq.cm. In contrast, the anodic current density of the sample welded without purification is 20,000 uA/sq.cm at the reversing potential. For the 5556 GMA welds made with helium shield gas only, the anodic current densities for both types of samples are relatively higher. However, the trend in the difference observed is unaltered. The anodic current density for the sample welded with purification at the reversing potential is 10,000 uA/sq.cm, which is still less than the 15,000 uA/sq.cm. exhibited by the sample welded without purification.

Although both the anodic current density and the free corroding potential are relatively lower in the coupons made with purified gas, the coupons nevertheless actively corrode. The corroded surfaces are illustrated in figures 43a and 43b. It was surprising to note that even the sample which appeared the best in terms of surface cleanliness did not show any degree of relative improvement in corrosion properties. It should be noted, however, that this

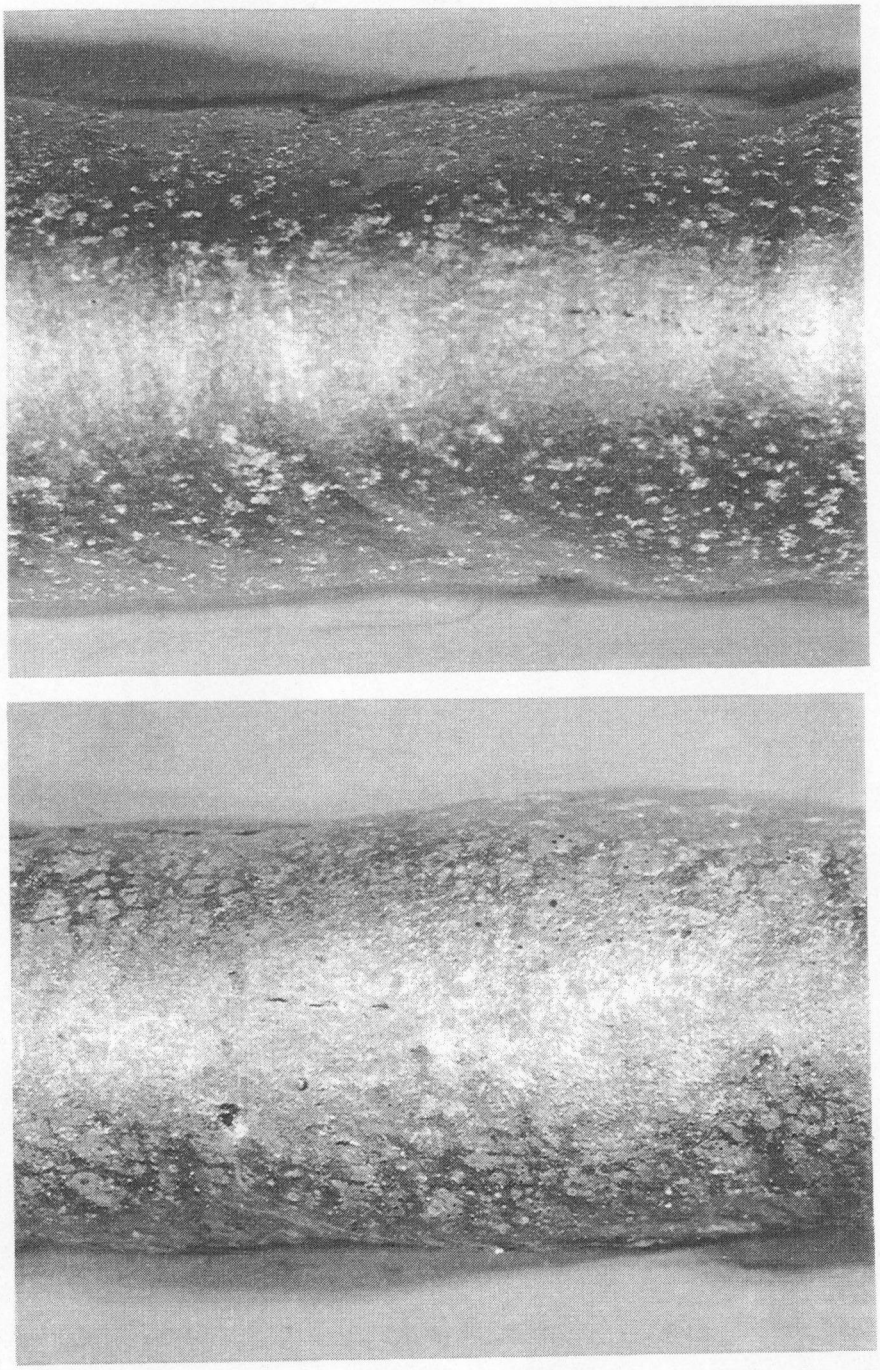


(a)



(b)

Figure 42. Anodic polarization scan of 5086 GMA welds in 3.5% NaCl solution. (a) welds shielded with purified and contaminated helium gas (b) welds shielded with purified, and moisture -and-oxygen contaminated 65argon-35 helium shield



(a)

(b)

Figure 43. Weld surfaces of GMA welded 5086 aluminum after electrochemical corrosion testing in 3.5% NaCl solution, indicating severe pitting
(a) Nanochem purified helium shield (b) Moisture contaminated helium shield

behavior is only relevant to the specific test environment chosen. Different test solutions produce different corrosion resistance characteristics. The test medium, in this case a 3.5% wgt. NaCl solution, was chosen because of its similarity to sea water and its relevance to shipyard fabrication alluded to earlier. Actual welds are therefore not recommended for service in strong chloride solutions (pH<3) without protective paints or cathodic protection measures.

6.7.5.2. **1% Deareated Sodium Sulphate Tests**

Following the active corrosion observed in all coupons in the 3.5 % NaCl solution, electrochemical anodic polarization tests were repeated using 1% sodium sulphate deaerated solution as the test medium. This chloride-free, near neutral (pH=7.2) medium is less aggressive than the 3.5% NaCl medium used in the previous tests. The results of the anodic polarization scans are indicated in figure 44. A significant difference can be observed in the corrosion characteristics of the two test samples. The passivation characteristics of the sample welded with purified helium are very strong. As seen in the scan of the sample welded with repurified helium, a passive film associated with a current density of 80 uA/cm^2 was rapidly established at the free corroding potential (-900 mV). This film continued to remain stable, (increasing only gradually in current density) even at potentials as high as 2600 mV, with no breakdown. At this point, the potential was intentionally reversed to initate the downscan and final segment of the test.

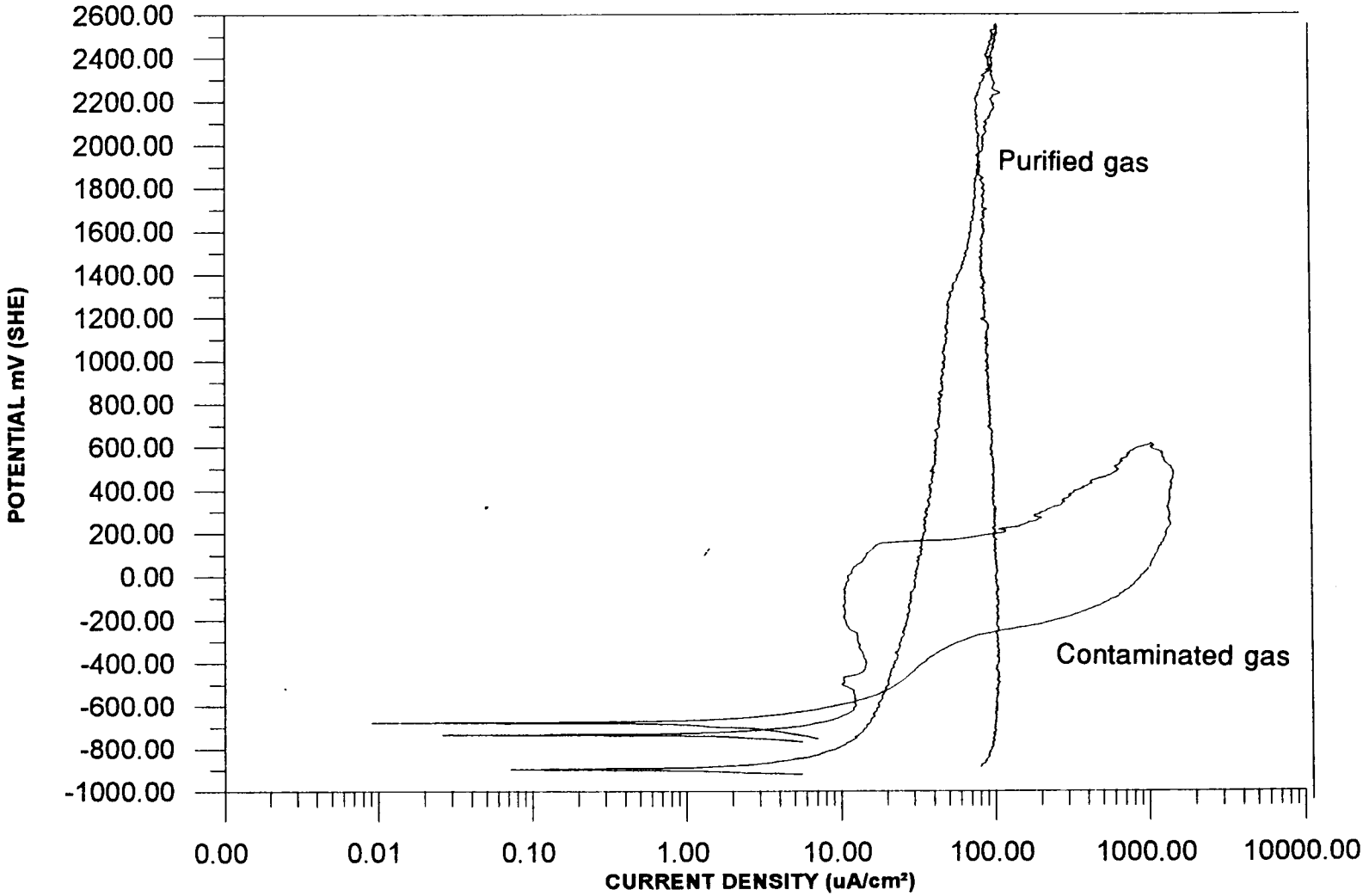


Figure 44. Anodic polarization scan of GMA welded 5086 aluminum, (shielded with purified and moisture contaminated helium) in 1% deaerated sodium sulphate solution

In contrast to this behavior, the sample welded with contaminated gas experienced relatively very little passivation, with passive film breakdown starting as early as 200 mV. Thereafter, the anodic current density kept rising rapidly, indicating active corrosion till the threshold current density of 1000uA/cm^2 was reached. The potential at this programmed threshold current density was 2400 mV less than the 2600 mV achieved by the purified gas sample at its maximum current density of 90uA/cm^2.

The significant difference in the polarization characteristics is attributed to the nature of the oxide films prevalent on the as-welded surfaces. In samples welded with purified gas, the oxide films were smooth and defect-free. The welds were shiny and the integrity of the oxide was responsible for strong passivation characteristics. In the samples welded with contaminated gas, the surfaces were laden with oxide characterized by minute defects, porosity, soot, and overall roughness. This type of a film with many surface defects resulted in the natural loss of passivity. The consequent infiltration of the test soultion into the defects and flaws on the surface led to active corrosion of the underlying metal. This result points to the fact that for welds in chloride-free, neutral type soultions, the effect of shield gas purification is clearly positive.

6.8. **6061 GMA Weld Tests**

6.8.1. **Surface Appearance**

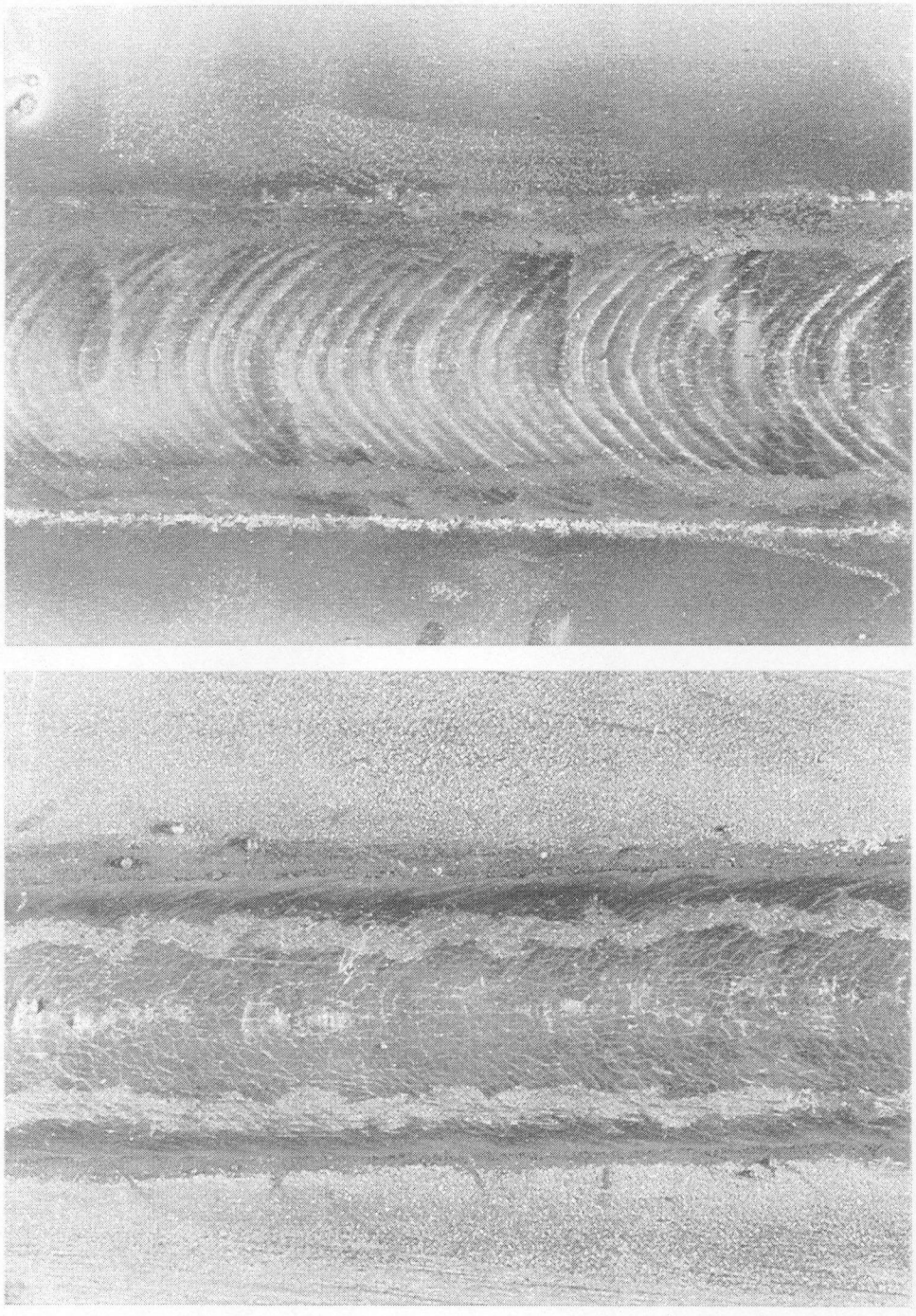
Unlike in the previous GTA and GMA tests conducted on 1100 and 5086 aluminum, the coupons welded with and without purification of contaminated

gas were not strikingly different in appearance. However, an overall difference in luster and oxide cover was evident. Figure 45a depicts the surface of a sample welded with Nanochem-purified helium gas, which was originally contaminated with approximately 300 ppm moisture. Figure 45b depicts the surface of a sample welded with moisture-contaminated gas. The degree of oxidation is strongly apparent in the snakeskin-like appearance caused by the cracks in the oxide coating. As opposed to this dull, flaky-looking surface, the sample welded with Nanochem depicted earlier appears clean and shiny. The sample welded with bottled helium, on the other hand, is depicted in figure 45c. When the bottled helium was passed through the Nanochem purifier, the resulting weld surface seemed to be slightly more shiny (figure 45d). This difference was confirmed using photography. The two pictures illustrated in figures 45c and 45d, were shot at identical camera settings using identical illumination lights. The slightly brighter appearance of the sample welded with bottled helium passed through the purifier can be noticed.

Analysis conducted to determine the impurities present in the bottled gas revealed the presence of 16 ppm oxygen and 1 ppm moisture in the gas. It is perhaps this impurity level that resulted in the slightly dull appearance of the samples welded with only the bottled gas.

6.8.2. **Bend Testing**

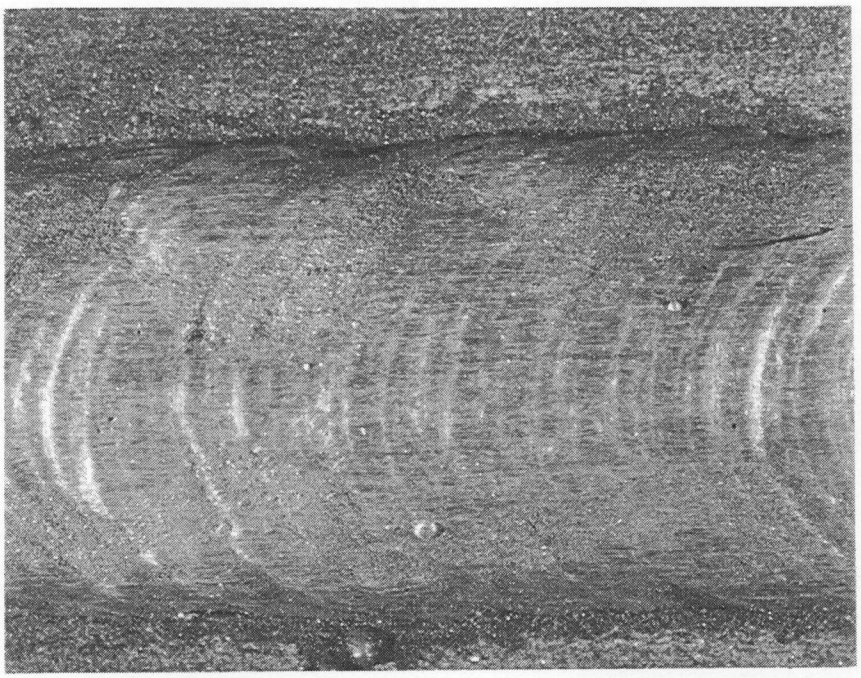
Two welds samples of each type (welded with contaminated and with purified gas) were tested for bend ductility as per ASTM E190 recommended test procedures. Since the weld coupons had been so selected to match the



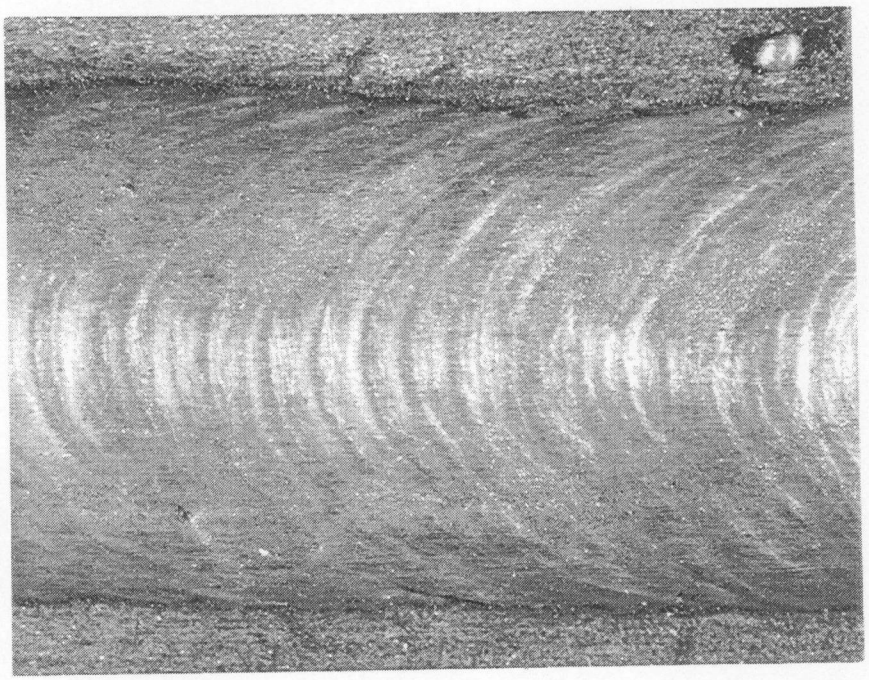
(a)

(b)

Figure 45. Effect of shield gas purification on the surface appearance of GMA welded 6061 aluminum. (a) Nanochem purified helium shield (b) moisture contaminated helium shield



(c)



(d)

Figure 45 (continued). (c) welding grade helium shield
(d) Nanochem purified, welding grade helium shield

prescribed specimen thicknesses for bend testing, no further machining was necessary. However, the two ends of each weld had to be ground flush with the base metal to maintain the 3/8" thickness at the end supports of the test jig. Longitudinal face bend tests were conducted. In other words, the test weld was parallel to the longitudinal axis of the specimen and was bent such that the face of the weld was the tension surface. The samples were carefully monitored for the initiation of any cracks during testing. The results of the bend testing are indicated in figure 46.

The bend angle was measured at the first sign of crack appearance. As seen in figure 46, the most significant difference was observed between the samples welded with contaminated gas and samples welded with purified gas. The contaminated gas samples tested failed consistently at a 20 degree bend angle. However, the welds made with the same gas after purification could be bent to 105 and 120 degree bend angles before crack initiation.

The results substantiated the fact that contaminated shield gas not only produces welds that are poor in appearance but also poor in mechanical properties. Another fact substantiated was that Nanochem purification of contaminated gas not only produces clean and shiny welds but also significantly improves the mechanical properties.

The poor ductility exhibited by the sample welded with bottled helium was surprising. As indicated before, even the surface appearance was not as shiny as the welds made after purifying the same gas. The analysis conducted for impurities on the bottled gas revealed that the bottled gas contained only 1 ppm moisture and 16 ppm oxygen. It is unlikely that the poor weld ductility is a

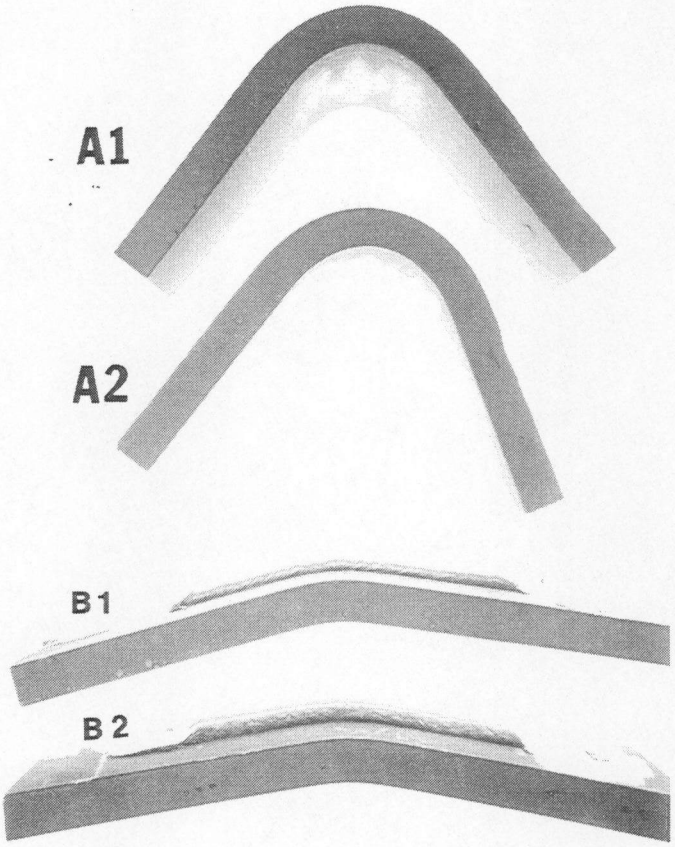


Figure 46. Appearance of GMA welded 6061 aluminum after bend testing till failure, indicating difference in ductility as a function of shield gas purity (Samples A1 and A2 were shielded with Nanochem purified helium; Samples B1 and B2 were shielded with moisture contaminated helium.)

result of this insignificant amount of impurity. Several explanations can be offered to explain the poor behavior of the welds made with bottled gas:

1. This set of welds was the first in the series of test welds made. Hence, there exists a possibility that sufficient moisture may have adsorbed onto the gas lines and gun parts which may have ingressed into the weld pool. The moisture in the lines could arise from atmospheric condensation during storage or condensation caused by cooling water used in the torch. Experiments in the literature have also reported that the first "cold" welds are usually characterized by unusual amounts of moisture desorbed from the tubing walls into the shield gas, which is subsequently eliminated due to a drying effect as further welding is continued [69].
2. Although not very likely, sudden air drafts may have also caused momentary loss of part of the shielding, allowing air to contaminate the surface of the molten pool.
3. Filler wire compounds are often responsible for causing weld pool contamination resulting in porosity. The reason that the weld wire surface has a high potential as a major source of contamination is that the ratio of wire surface area to volume of deposited weld metal is high. However, if the filler wire was the reason behind the degraded weld properties, the effects would have been seen in all the other weld coupons tested. Since this was not the case, the theory of weld contamination due to the filler wire is suspect.

In any event, the more important test results, namely those indicating a vast improvement in properties of welds made with Nanochem-purified gas, were the main focus of the study and these results were analyzed in greater detail.

6.8.2.1. **SEM Analysis of Bend Samples**

The bend samples indicating a major difference in ductilities (20 degrees for the samples welded with contaminated gas and 120 degrees for the samples welded with the purified gas) were sectioned to open up the crack produced at failure. The crack surfaces were then examined using scanning electron microscopy. The results were very conclusive as attested by figure 47 (a & b). The sample welded with the purified gas (figure 47a) is solid throughout the crack section except for a freak pore at the exact location of the weld failure, which is also the reason for failure in that particular weld. On the other hand, the crack surface of the sample welded with contaminated gas (figure 47b) is clearly riddled with porosity.

6.8.2.2. **Pore Count**

In order to quantify the approximate difference in the pore quantities, computerized image analysis was performed on the two surfaces. The analysis was conducted in the back scatter mode to facilitate greater definition of the pores (figures 48a and 48b). The analysis indicated that in the sample welded with Nanochem -purified gas (figure 48 a), the pore area in the crack section was a mere 10%. In contrast, as shown in figure 48b, the sample welded with contaminated gas was characterized by almost 50% porosity. Evidently, this "swiss cheese" characteristic of the weld qualifies it as an extremely poor weld.

The explanation for the presence of porosity is straightforward. Moisture present in the helium dissociated into hydrogen and oxygen in the welding arc.

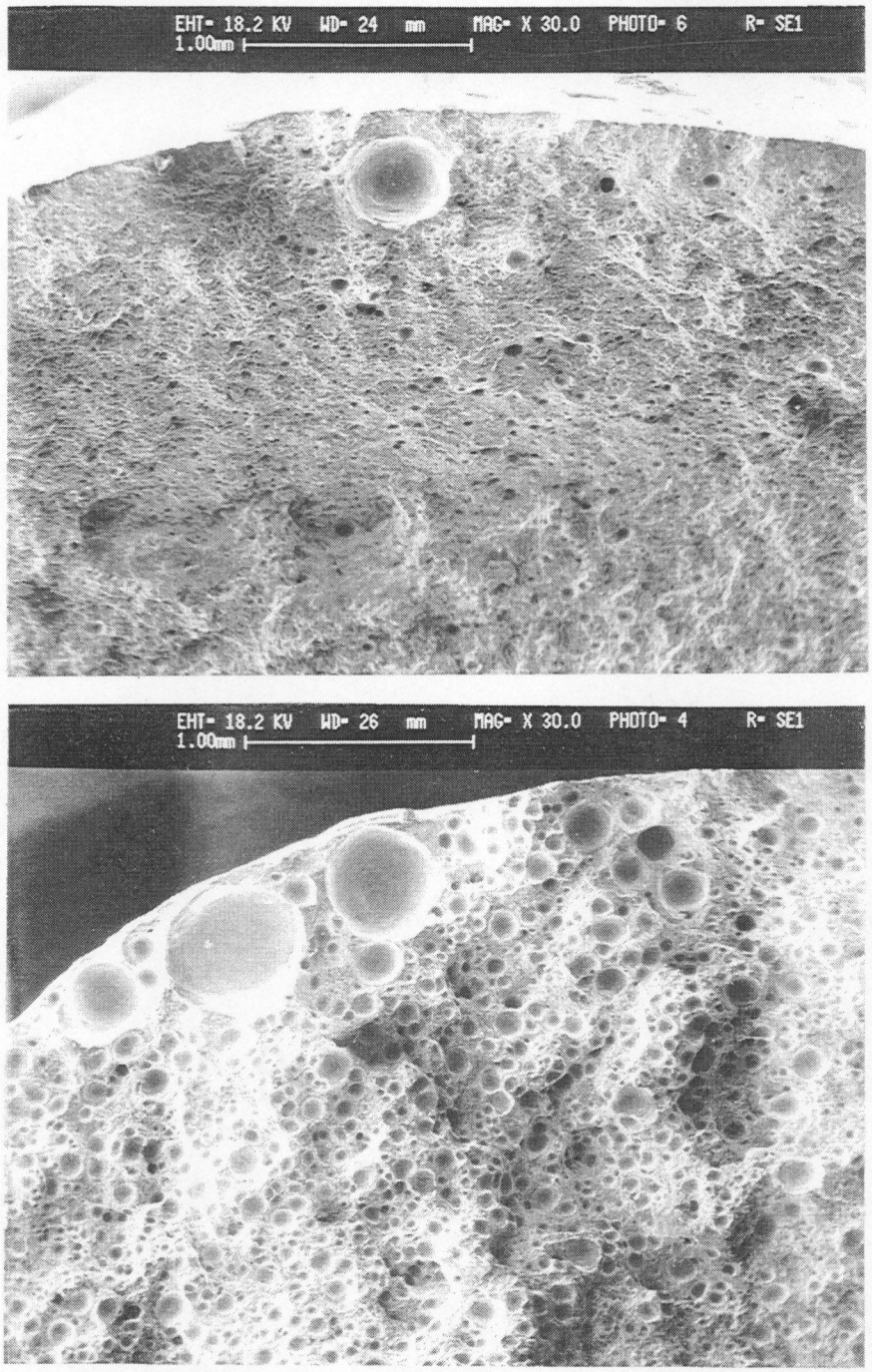


Figure 47. SEM analysis of crack tip of bend test sample.
(a) weld shielded with Nanochem purified helium gas
(b) weld shielded with moisture contaminated helium gas

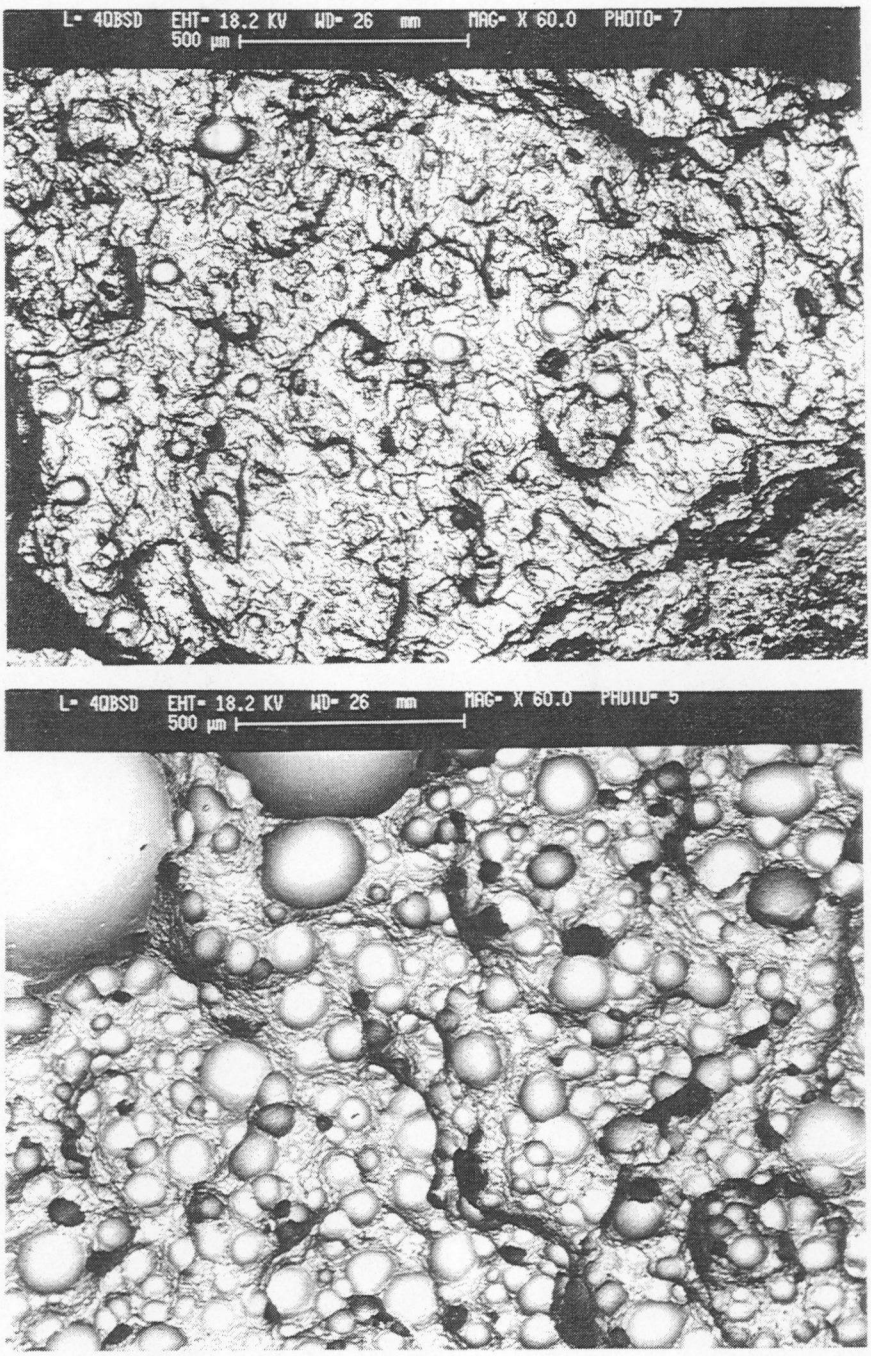


Figure 48. Backscattered image of crack tip of bend test sample used for image analysis. (a) weld shielded with Nanochem purified helium gas - 10%pore level (b) weld shielded with moisture contaminated helium gas - 50% pore level

The oxygen was mainly instrumental in generating the dross seen on the surface of the sample. The hydrogen was responsible for the porosity. It is well established that although molten aluminum can readily dissolve hydrogen, it can hold little hydrogen at room temperature. Therefore, hydrogen is rejected as the weld pool freezes. However, if the cooling rate of the weld puddle is fast enough, the hydrogen cannot rise to the surface but freezes within the weld metal, causing weld porosity.

The important issue after having determined that shielding gas purity largely affects porosity is to speculate on what kind of an effect pores have on aluminum welds. Porosity in aluminum alloys can be tolerated to some extent before there is any effect on the mechanical properties. As proven in these tests, bend ductility is definitely lowered. Tensile properties are also reduced when the amount of porosity is high.

6.8.3. **Corrosion Resistance**

Previous corrosion testing performed on GMA welded 5086 aluminum had indicated that in near neutral sodium sulphate medium, the passivation characteristics of contaminated gas welds were seriously degraded as opposed to the passivation characteristics of Nanochem-purified gas welds. The same test was repeated using 1% sodium sulphate deaerated solution. The results of the anodic polarization scans are indicated in figure 49. A significant difference is observed in the corrosion characteristics of the two test samples. The difference is similar to the test results obtained with the 5086 GMA welds. The passivation characteristics of the sample welded with purified helium are clearly

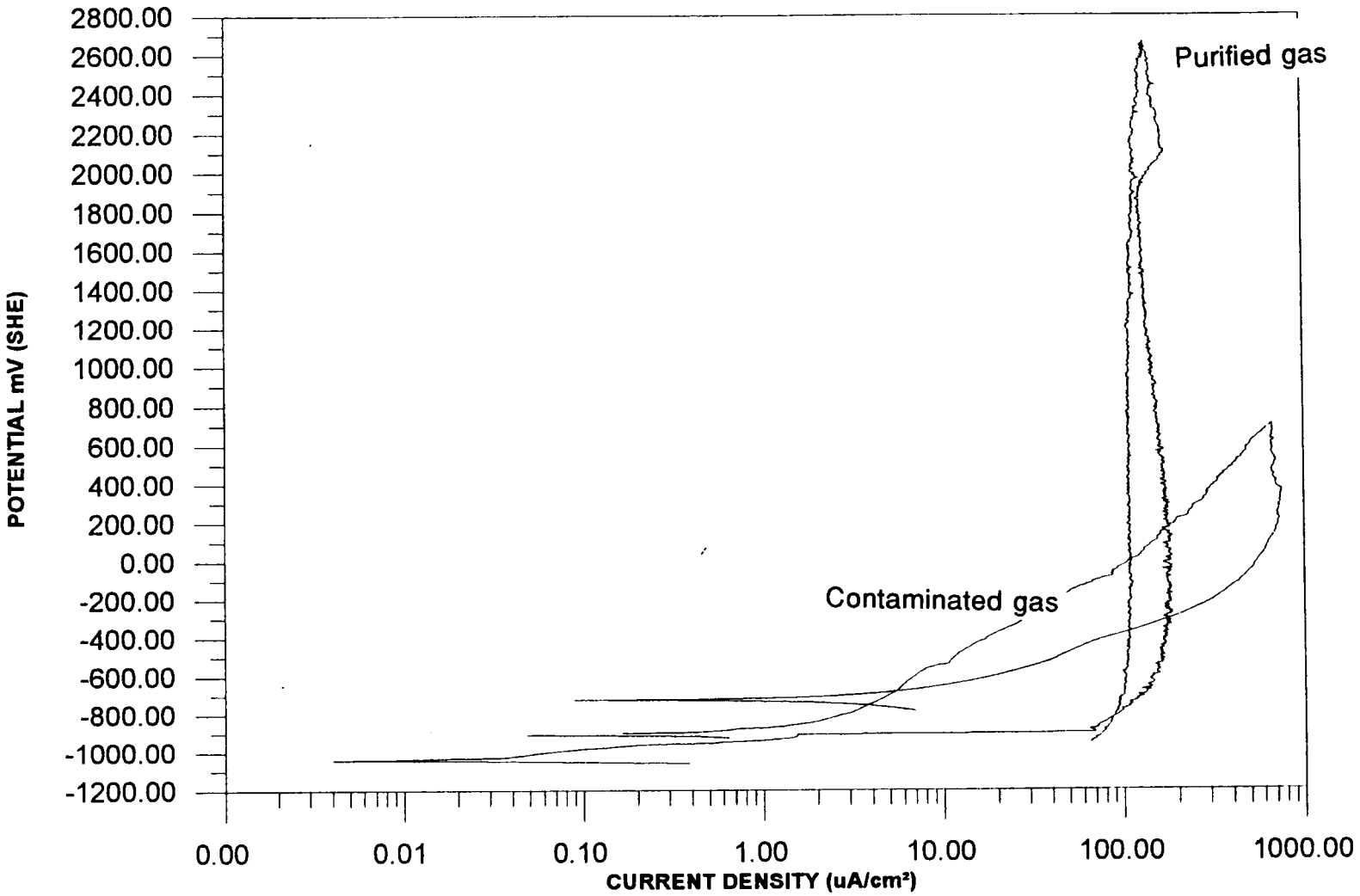


Figure 49. Anodic polarization scan of GMA welded 6061 aluminum GMA welds in 1% deaerated sodium sulphate solution

very strong. A passive film associated with a current density of 80uA/cm^2 was rapidly established at the free corroding potential (-1000 mV) in the sample welded with repurified helium. This film continued to remain stable even as high as 2600 mV, with no breakdown. At this point, the potential was intentionally reversed to initate the downscan and final segment of the test.

In contrast to this behavior, the sample welded with contaminated gas experienced active corrosion, with absolutely no indication of the formation of a passive film. Even the free corroding potential to start with was 400 mV less noble than the sample welded with repurified gas. Thereafter, the anodic current density kept rising, indicating active corrosion, and soon the threshold current density (1000uA/cm2) was reached. The potential at this programmed threshold current density was a mere 400 mV, compared to the 2600 mV achieved by the purified gas sample, without even exceeding 80uA/cm2 current density.

As with the 5086 GMA welds, the difference in the corrosion behavior is attributed to the difference in the quality and integrity of the weld zone oxide produced with and without shield gas purification. The fragmented, flawed oxide on the contaminated gas welds was responsible for the poor corrosion characteristics observed as opposed to the Nanochem-purified welds.

6.9. AL-6XN Alloy: GTA Weld Tests

6.9.1. Surface Appearance

The importance of visual examination of a weld as a means to test welds cannot be undermined. Visual inspection, especially in a high volume production setup, is often the only test performed by the quality inspector. Weld defects can often be so glaringly apparent that they are recognized immediately. In the experiments performed, visual inspection provided the first conclusive results almost as soon as the arc was extinguished.

6.9.1.1. Fusion Zone

For the coupons welded using identical parameters with and without shield gas purification, very obvious differences were found in the appearance of the welds. The effects of extreme gas contamination are displayed in figure 50a. The sample shown was welded with argon gas containing 434 ppm moisture and 196 ppm oxygen. The fusion zone is grossly contaminated, with clear evidence of severe oxidation. The weld is also characterized by a wide heat tint region surrounding the fusion zone. The same contaminated gas, when passed through the Nanochem purifier, resulted in a weld whose appearance is documented in figure 50b. The contrast between the welds made with and without purification is striking. As opposed to the severely oxidized, lusterless weld made with contaminated gas, the "purified-gas" weld is shiny and totally free of any heat tint. In fact, the fusion zone is so clean that

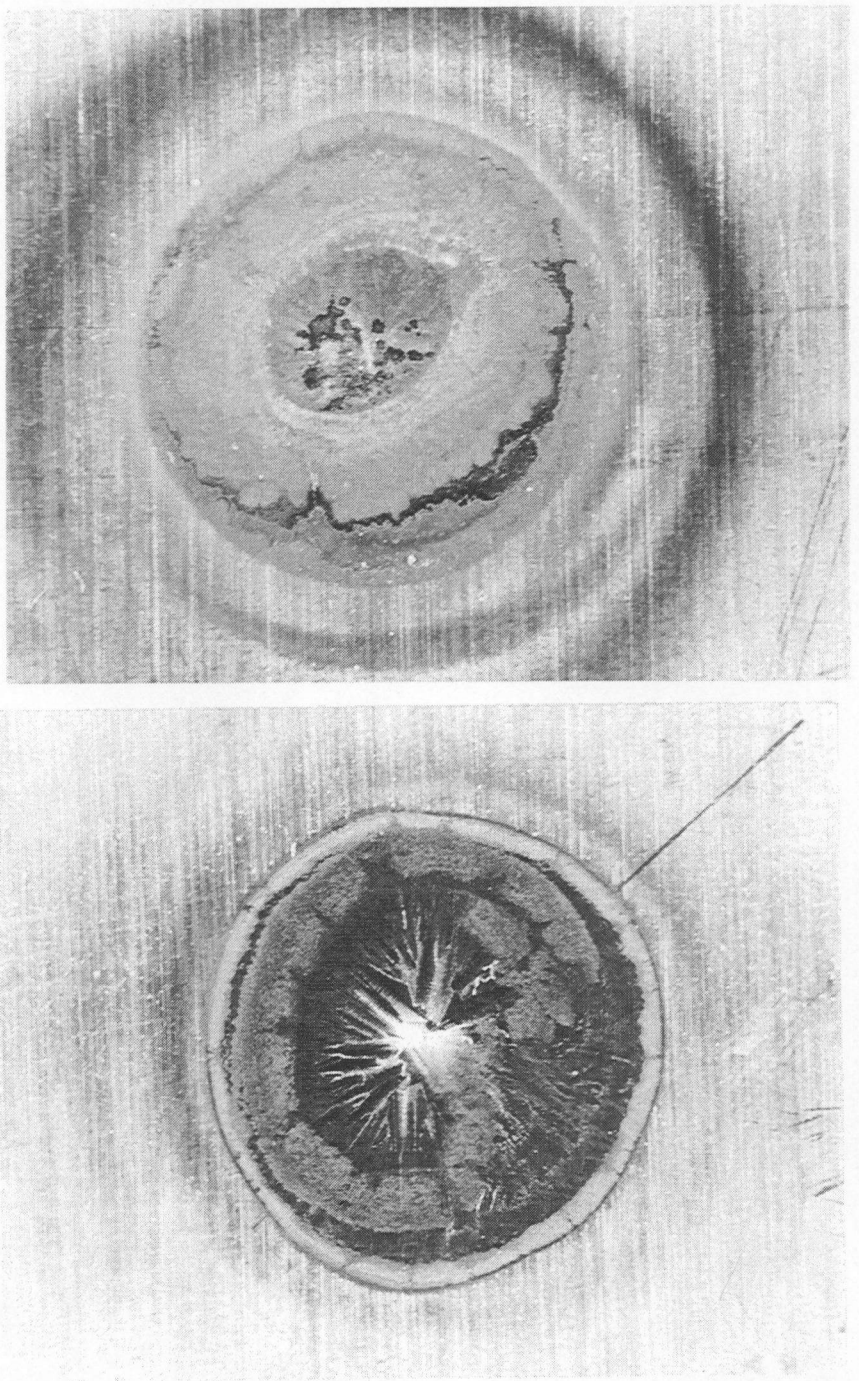
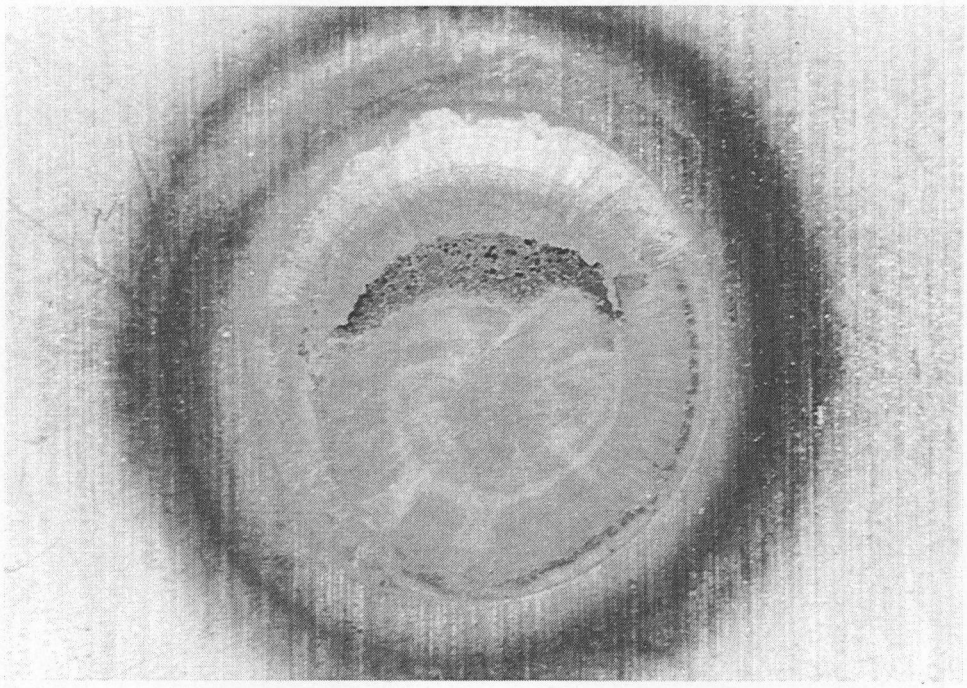
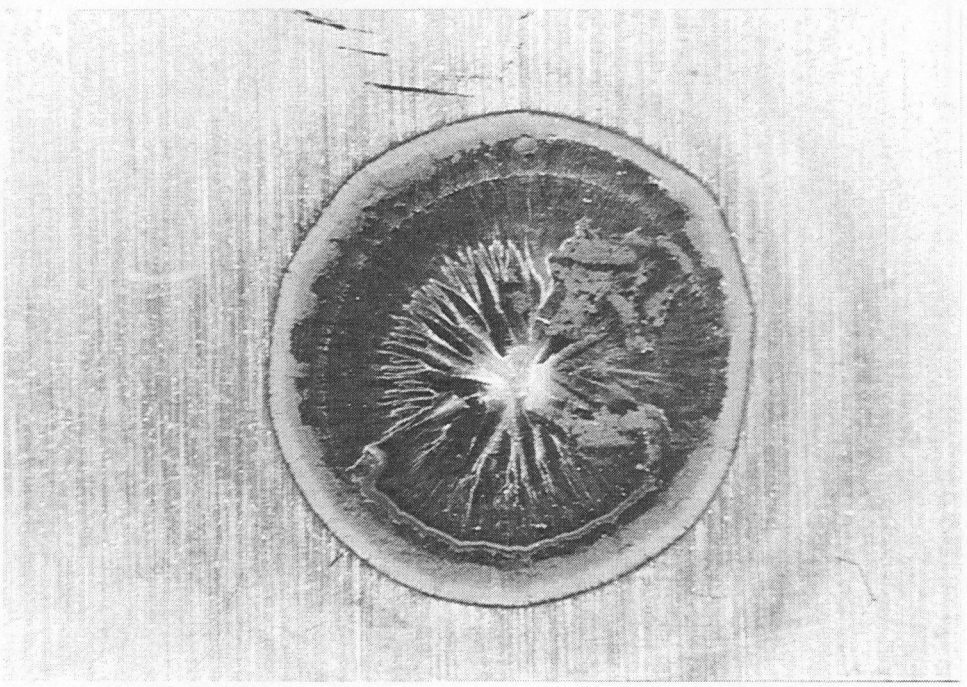


Figure 50. Effect of shield gas purification on the surface appearance of GTA spot welds of AL-6XN alloy. (a) argon shield contaminated with oxygen (196 ppm) and moisture (434 ppm) (b) Nanochem-purified argon shield



(c)



(d)

Figure 50 (continued). (c) argon shield contaminated with 8 ppm oxygen and 3 ppm moisture (d) Nanochem purified argon shield

even the solidification substructure-equiaxed columnar dendritic in the weld center and cellular dendritic elsewhere in the weld is clearly visible.

Typical weld appearances at lower gas contamination levels (8 ppm moisture and 3 ppm oxygen) are presented in figures 50c and 50d, welded with and without purification, respectively. In general, the welds made with purified gas remained consistently shiny irrespective of the amount of contaminant originally present in the gas before purification. Stereo microscopy revealed that the few oxide patches present were mostly concentrated near the periphery of the fusion zone. However, with increasing contamination, fragmented oxides were increasingly found towards the weld center.

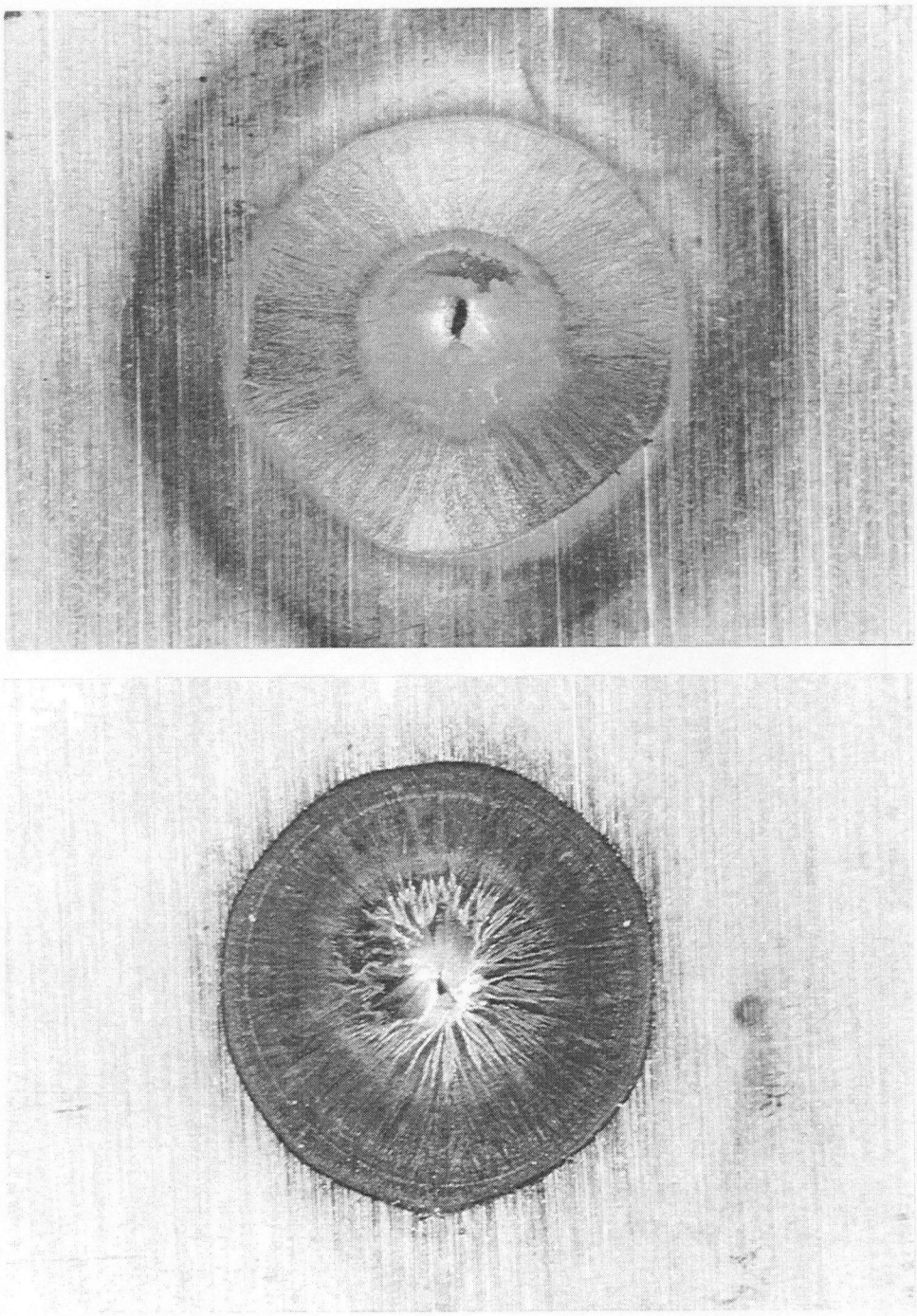
For the samples welded without gas purification, the fusion zone was consistently oxidized, and the solidification substructure remained completely obscured even with low amounts of contaminants (2 ppm oxygen and 50 ppm moisture). Isolated patches of small pores were also observed in some samples. The fusion zone diameter was also slightly irregular in comparison to the welds made with purified gas. This difference suggests that the molten pool flow is influenced by the oxygen and moisture dissociated in the arc. The dissociated contaminants influence either the surface tension of the molten puddle or the arc stability. This effect is manifest in the irregularity of the fusion zone periphery as opposed to the more circular periphery of the welds made with purified gas.

No differences, however, were found between the samples welded with bottled argon and Nanochem-purified argon, indicating that the regular bottled argon used was of satisfactory purity.

The features observed on the underside/root of the welds were similar to the features observed on the face of the welds shielded with and without purification of the shield gas. The root surfaces were again markedly different as illustrated in figures 51a and 51b. (The welds were welded with shielding gas contaminated with 25 ppm oxygen and 66 ppm moisture.) The backing gas used was tapped from the main shielding gas line that supplied the torch gas to shield the face of the samples. In effect, the composition of the gas used for shielding the face and root surfaces of the welds were the same. This fact explains the similarity in surface topography and cleanliness of the face and root surfaces. However, the degree of oxidation observed on the root surface of the contaminated gas welds was found to be less than that seen on the face surface. For the weld made with purified gas, a complete absence of oxide was observed. This finding was consistent in all welds.

6.9.1.2. **Heat Tinted Area**

In the region adjacent to the fusion zone and within the gas coverage zone, the most striking feature observed was a multicolored heat tint in the welds made using contaminated shield gas. Depending on the temperature experienced by that region, different colors were observed around the weld area, including dark and light yellow, light blue, purple and amber. For the welds made with purified gas, only faint brown colorations were observed in some samples. The colored areas can be examined in the figures pertaining to the previous section.



(a)

(b)

Figure 51. Effect of shield gas purification on the root surface appearance of GTA spot welds on AL-6XN alloy. (a) argon shield contaminated with 66 ppm moisture and 25 ppm oxygen (b) Nanochem purified argon shield

The regions near the surface of a heat-tinted alloy can be significantly depleted in chromium. The defects, internal stresses, and chemical composition of the heat-tint oxide make it a poor barrier to any corrosive media. The chromium-depleted layer of base metal below the heat tint is less resistant to localized corrosion than the base metal in certain environments such as oxidizing chlorides. Pits initiated in the chromium-depleted surface layer can propagate through sound base metal. The corrosion of the heat tinted oxide can also activate the metal beneath it in a manner similar to free iron particles on a surface. In light of this information, the creation of heat tint by contaminated gas and the beneficial effect of Nanochem purification in terms of preventing the formation of heat-tint was obviously a promising find. The exact effect in terms of corrosion behavior was quantified using electrochemical corrosion tests. The details of this study are addressed in the sections detailing corrosion studies.

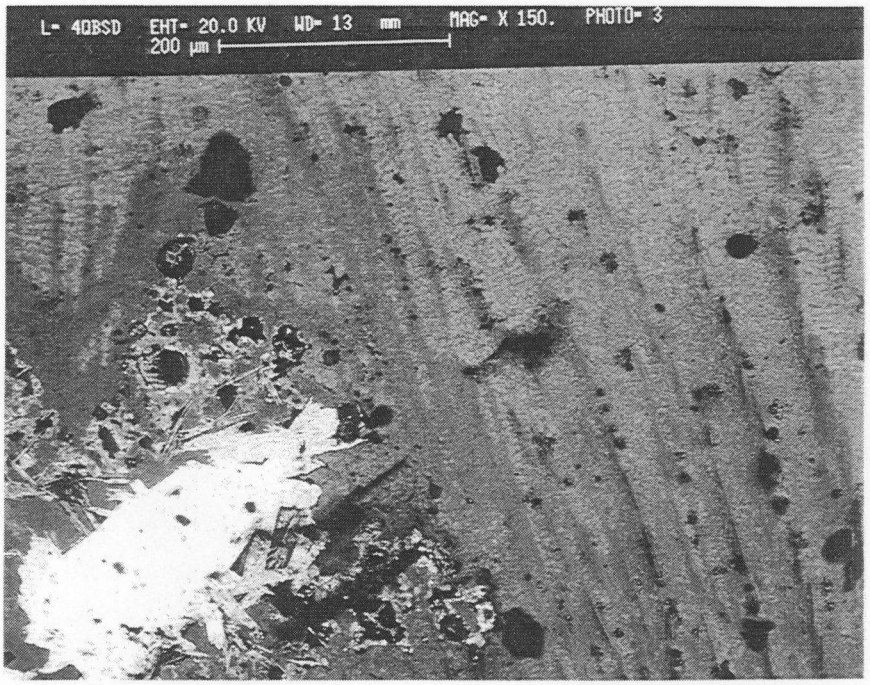
6.9.1.2.1. **SEM Studies**

In order to detail the differences in the surface topography and possible compositional changes of the samples welded with and without purified gas, scanning electron microscopy studies were conducted. Unfortunately, the oxide content cannot be quantified using EDS in conjunction with SEM. Hence, exact quantification of oxidation could not be accomplished. However, other interesting results were generated. Lanthanum-and cerium rich-oxides were found distributed on the surfaces of samples welded with and without purification. The oxide morphology and distribution also clearly varied in the

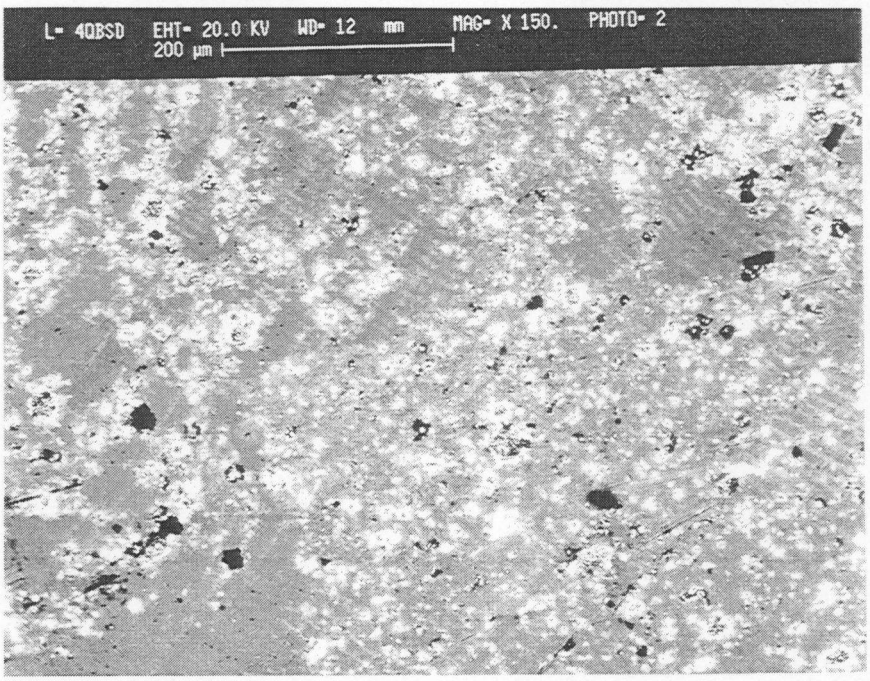
two types of samples. Figure 52a shows the oxide distribution in the sample welded with purification. As opposed to this typical appearance, figure 52b depicts the oxide on the surface of the weld made with contaminated gas. The oxide is clearly more fragmented and covers the entire field of view. EDS analysis (figures 53a and 53b) revealed that the chemical composition of the oxide lumps are essentially the same. The lanthanum and cerium present provides reason to believe that rare earth additions are made in the fabrication of the AL-6XN alloy. Misch metal is known to be used for deoxidation of basic electric or open hearth steels. The hot workability of austenitic steels running 4 to 40% nickel is also changed so that scrap losses are reduced by adding up to 0.2% rare-earth metals. There also exists a possibility that lanthanum and cerium additions are made to either the ceramic cup and/or the tungsten electrode, which together contributed to the oxidation observed. The extent of oxidation on the surface of the AL-6XN fusion zone surfaces is clearly linked to the moisture and oxygen present in the gas. The sample welded with purified gas shows a cleaner surface with only a single chunk of oxide. On the other hand, the sample welded with contaminated gas clearly shows relatively smaller oxide particles dispersed throughout the surface of the sample.

6.9.2. **Metallographic Examination**

Eight representative test coupons were metallographically prepared to examine the microstructural characteristics of welds made with purified gas and contaminated gas. Samples were also cross-sectioned. The sample depicted in figure 54a was welded with purified gas that originally contained 300 ppm

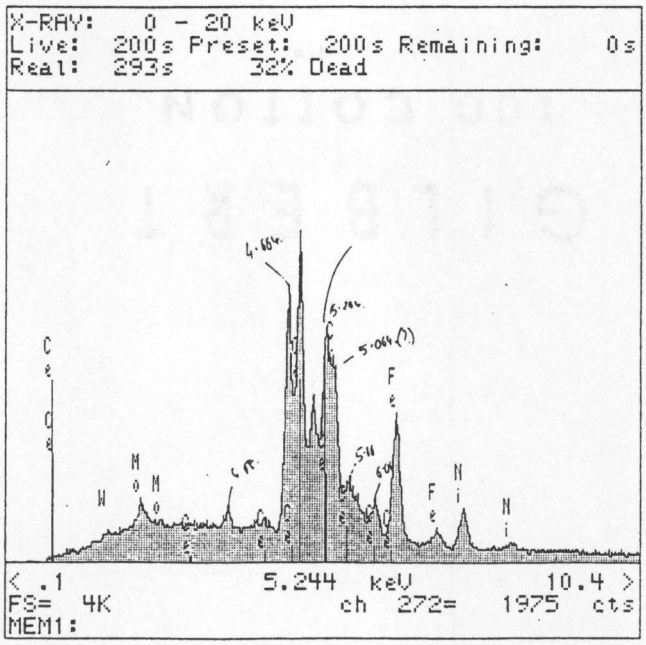


(a)

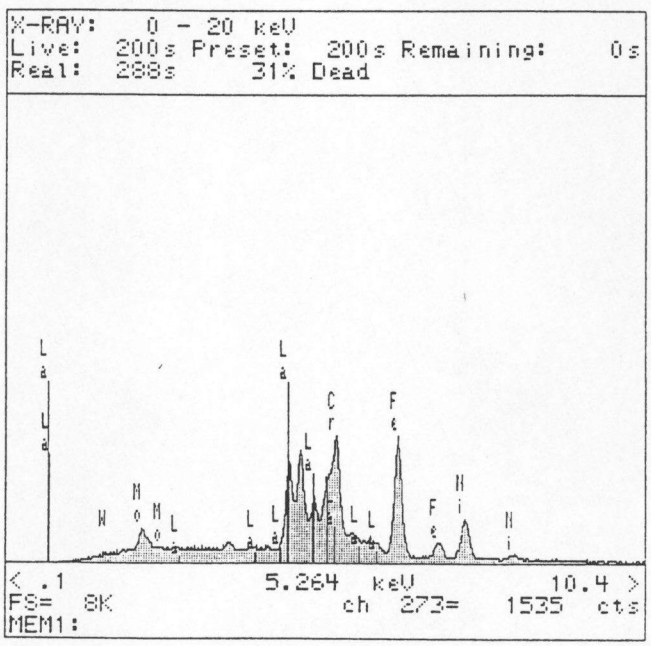


(b)

Figure 52. Oxide morphology and distribution on the fusion zone surface of GTA spot welded AL-6XN alloy. (a) Nanochem purified argon shield (b) moisture and oxygen contaminated argon shield



(a)



(b)

Figure 53. EDS analysis of fusion zone oxide of GTA spot welded AL-6XN alloy. (a) cerium oxide (b) lanthanum oxide

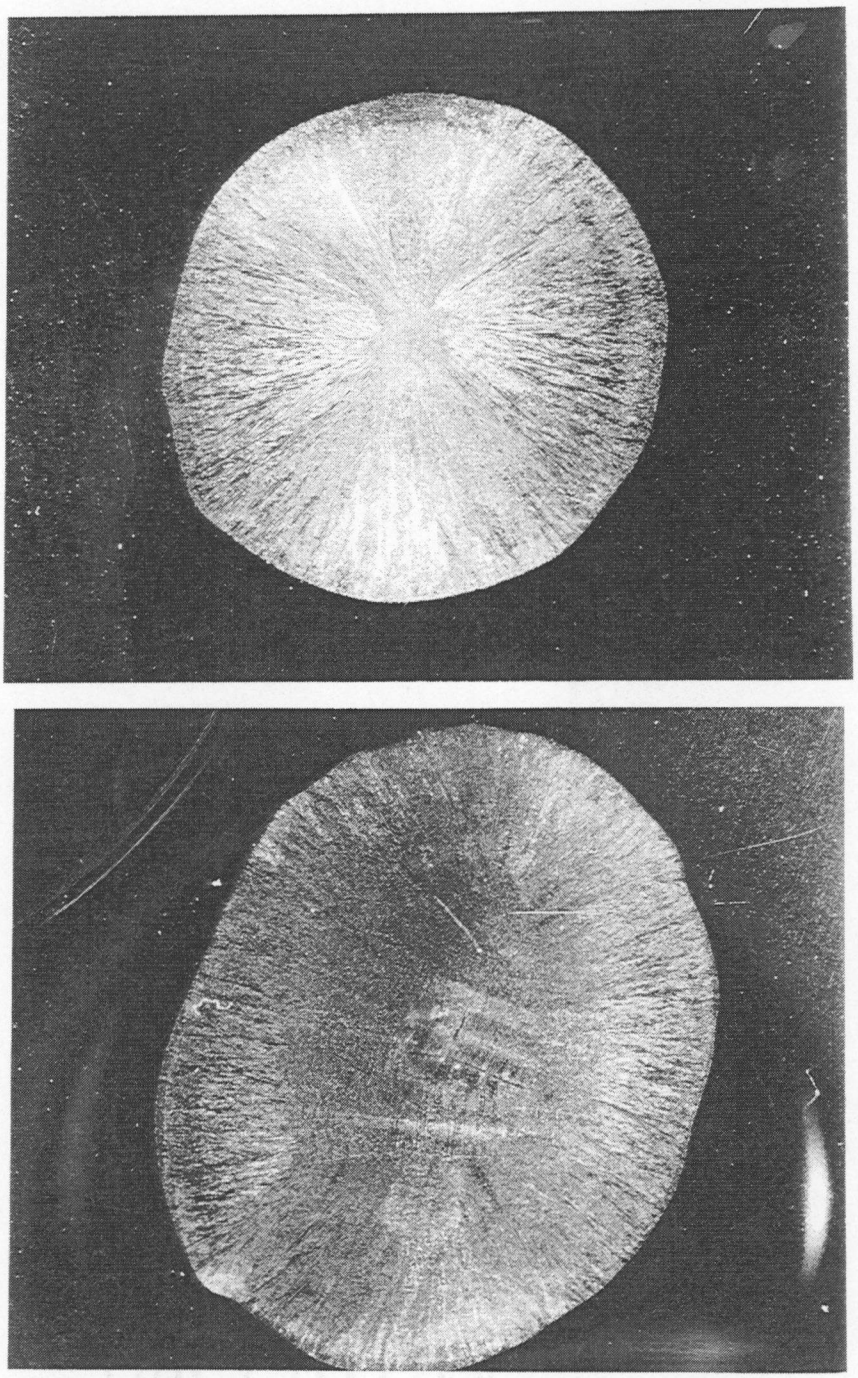


Figure 54. Macrostructure (600 grit) of AL-6XN GTA spot weld. (a) Nanochem purified argon shield (b) Argon shield contaminated with 100 ppm oxygen and 300 ppm moisture

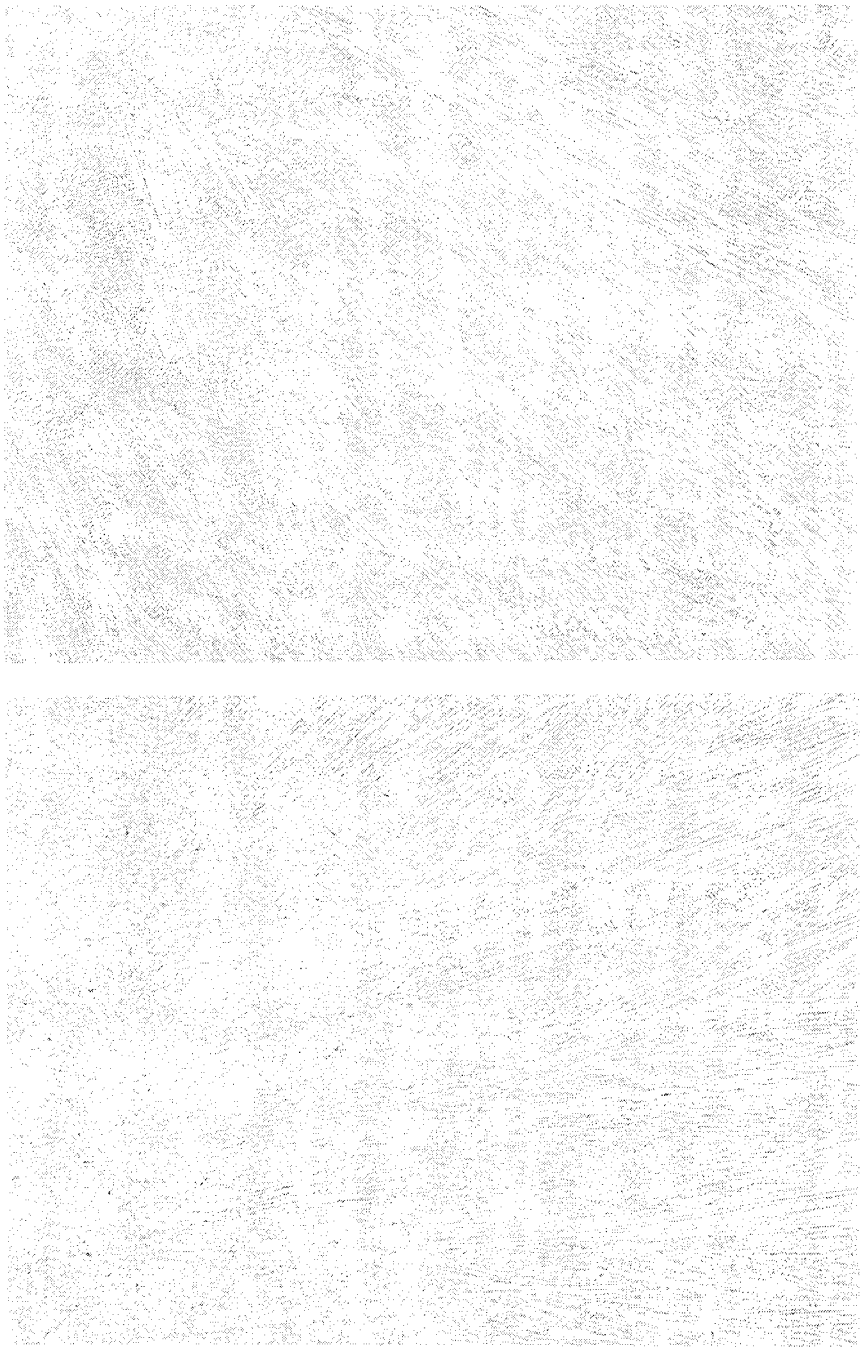
moisture and 100 ppm oxygen. As opposed to this, the sample welded with the same contaminated gas without any prior purification is depicted in figure 54b. Macroscopically, the differences are mainly apparent in the diameter of the weld spot. As alluded to earlier, the irregular peripheries of the "impure gas" samples as opposed to the near-circular peripheries of the "pure gas" samples are indicative of altered surface tension or instable arc behavior. Metallographically, however, no differences were observed. Figures 55a and 55b depict the microstructures of the samples described in figures 54a and 54b, respectively. The solidification substructure remained essentially the same. No differences in relative constituents, secondary phases, and microsegregation were observed. Hardness measurements also indicated no differences.

6.9.3. **Corrosion Testing**

The applications of AL-6XN, as alluded to before, are mainly in areas requiring corrosion resistance. The significant difference in appearance between contaminated gas and purified gas welds was speculated to have a major effect on corrosion resistance properties. Hence, in order to quantify this difference, several electrochemical and immersion corrosion tests were carried out. Testing was carried out in industrially relevant media listed below:

1. 6% ferric chloride
2. 10% sulphuric acid
3. 50% boiling sodium hydroxide

Some or all of the weldment zones were tested in the media listed above. The weldment zones tested were as follows:



(a)

(b)

Figure 55. Microstructure of AL-6XN GTA spot weld. (a) Nanochem-purified argon shield (b) argon shield contaminated with oxygen and moisture

1. As-welded surface
2. Ground fusion zone surface
3. Heat-tint area
4. Entire weldment (FZ, HAZ, BM)

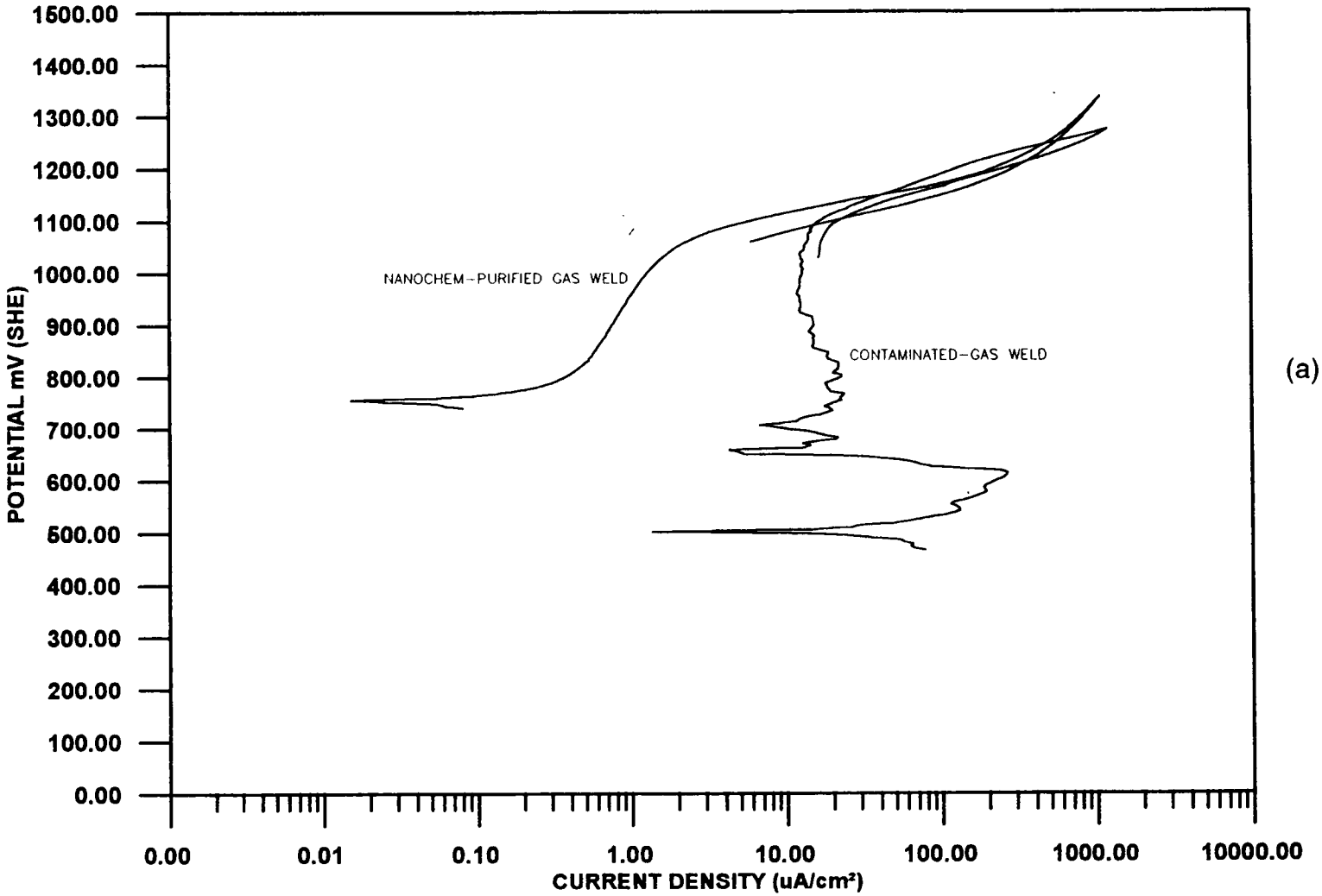
6.9.3.1. **6% $FeCl_3$ Immersion and Electrochemical Tests**

One of the most important characteristics of a stainless steel alloy exposed to solutions containing chlorides such as sea-water or flue-gas condensate is resistance to pitting and crevice attack. Electrochemical cyclic polarization tests were carried out in accordance with ASTM G-48 recommended procedures. The solution employed was 6% $FeCl_3$ at ambient temperature. Only the weld surface was tested for corrosion and the remaining portion of the weldment was masked with non-conductive paint. Two conditions were examined as follows:

1. As-welded fusion zone (welded with and without gas purification)
2. Ground fusion zone-600 grit (welded with and without gas purification)

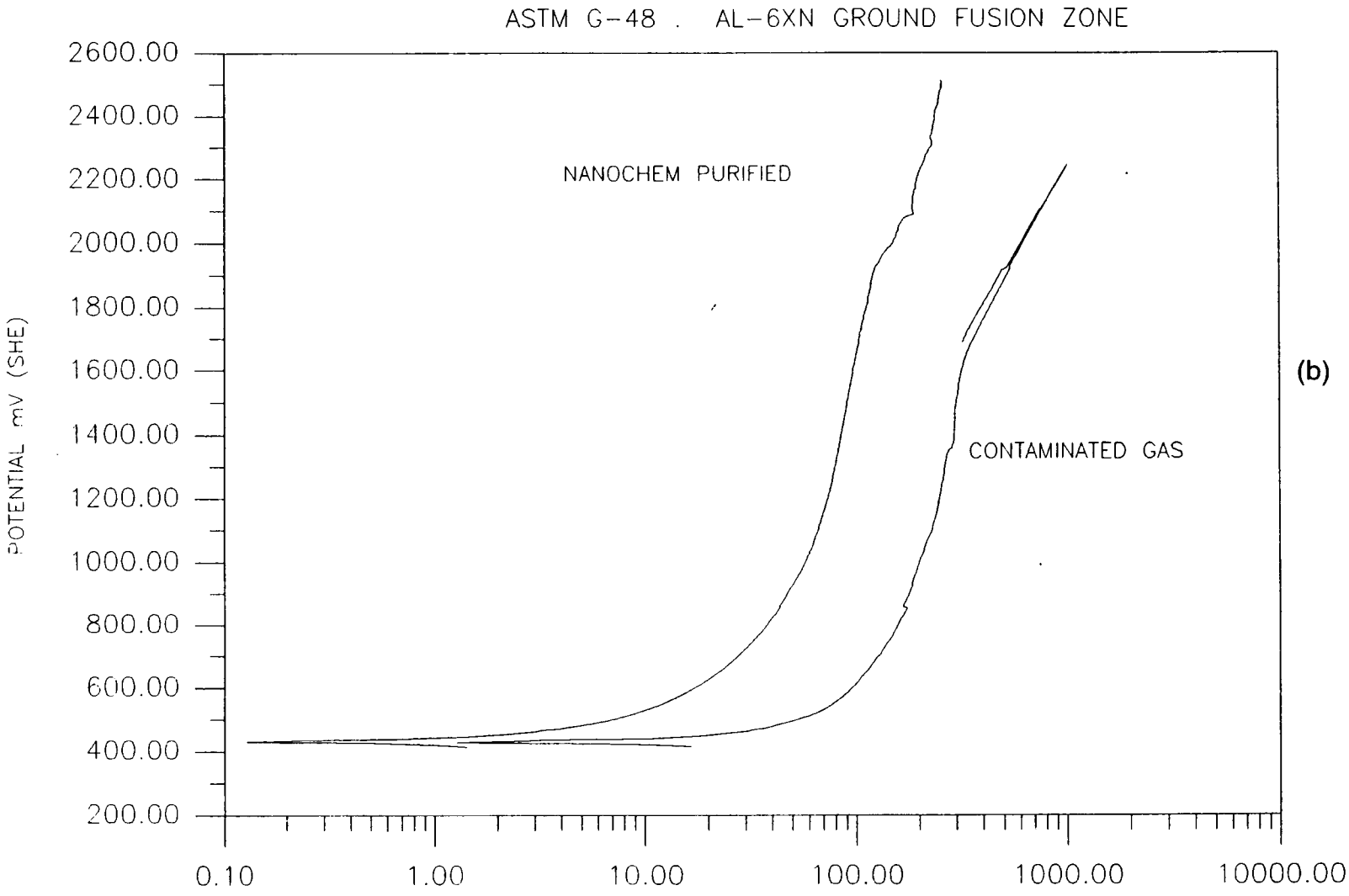
6.9.3.1.1. **As-Welded Fusion Zone**

The results of the tests showing the polarization scans conducted on the two samples in the as-welded surface condition welded with and without purification are depicted in figure 56a. The most apparent feature is that the sample welded without purification exhibits an overall current density that is higher than the current density of the sample welded with purification. The free



(a)

Figure 56. Anodic polarization scans of GTA autogenously welded AL-6XN alloy in 6% ferric chloride solution. (a) As-welded fusion zone



(b)

Figure 56 (continued). (b) 600 grit polished fusion zone

corrosion potential (E_{corr}) is also 200 mV less noble than the E_{corr} value of the sample welded with purification. However, it does show a clear indication of the formation of a passive film between 600 and 1100 mV (SHE). Such a passive film is not clearly indicated on the sample welded with purified gas. The passivation detected in the sample welded with contaminated gas may in fact be due to the oxide formed during welding itself. This speculation is reinforced by the noise/fluctuations seen in the anodic current density in the passivation range. These current fluctuations are caused by the local current variations on the oxide-laden, non-homogenous weld surface. Such fluctuations are not seen in the polarization scan of the sample welded with purification. Instead, the current associated with the formation of the passive film is found to increase gradually. At 1100 mV, the anodic current density increases rapidly for both samples. Some insignificant pitting may have occurred, as indicated by the slight hysterisis detected in the reverse scan. Any pits that may have formed are eventually healed/repassivated. The repassivation is indicated by the intersection of the reverse scan with the up-scan within the passivation zone.

6.9.3.1.2. **Fusion Zone (Ground)**

In the experiment described above, the surfaces of the weld test specimens were used in the as-welded condition. Hence, the test slightly deviated from prescribed ASTM G-48 pitting corrosion testing norms. It was only logical to run a second set of experiments employing a 600 grit ground fusion zone for testing. The results of this experiment are indicated in the scans presented in figure 56b. The free corroding potentials of both samples, welded

with contaminated gas and purified gas, are essentially identical (400 mV). Both samples seem to passivate, but at an extremely low rate, as attested by the very gradual rise in the anodic current density. Closer inspection reveals that this rise in current is 100 uA/cm2 over 2200 mV for the sample welded with purification and is 1000 uA/cm2 over 1800 mV for the sample welded with contaminated gas. In fact, the sample welded with purified gas did not reach the threshold current density of 100 uA/cm^2, even when the test was terminated at 2500 mV. On the other hand, the sample welded with contaminated gas reached the threshold current density of 100 uA/cm^2 at 2200 mV. The overall anodic current density for the sample welded without purification is larger than 100 uA/cm^2, (regarded as a high corrosion rate), while that for the sample welded with purification is less than 100 uA/cm^2 (regarded as a medium corrosion rate). Although not quantitatively significant, this observation does imply that the sample welded without purification corrodes more than the sample welded with purification. The surface appearance of the 600 grit polished sample before corrosion testing is presented in figure 57a. Observation of the samples after testing indicated that active, uniform corrosion had taken place evident in the etching effect seen in figure 57b.

6.9.3.1.3. **ASTM G-48 Immersion Tests**

Four welded coupons were tested using ASTM practice G-48 as the basis for immersion corrosion testing. The test parameters used for the corrosion tests were as follows:

Solution: Ferric chloride 6%

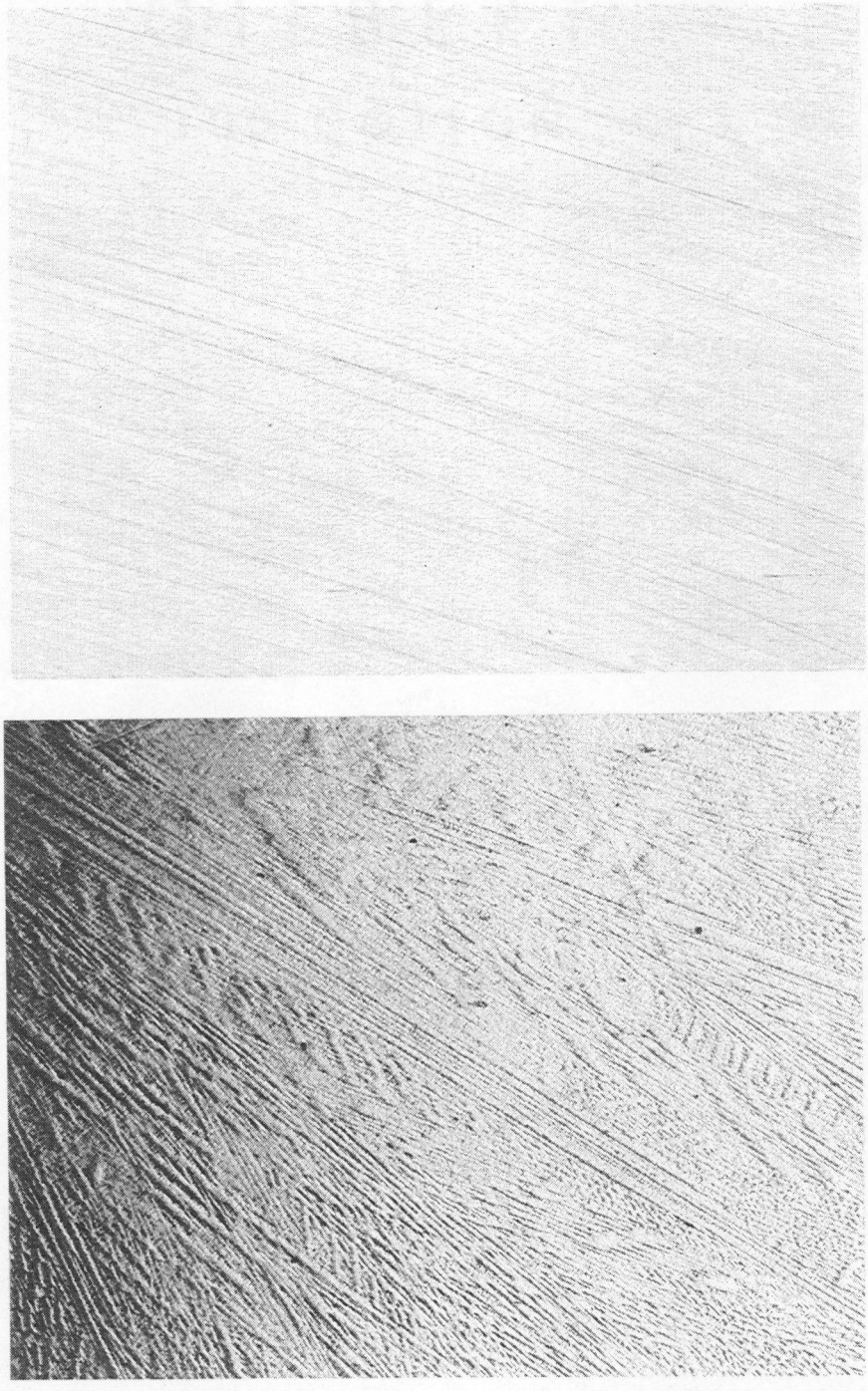


Figure 57. Appearance of GTA welded AL-6XN ground fusion zone before (a) and after (b) electrochemical testing in 6% ferric chloride solution

Time: 48 hours

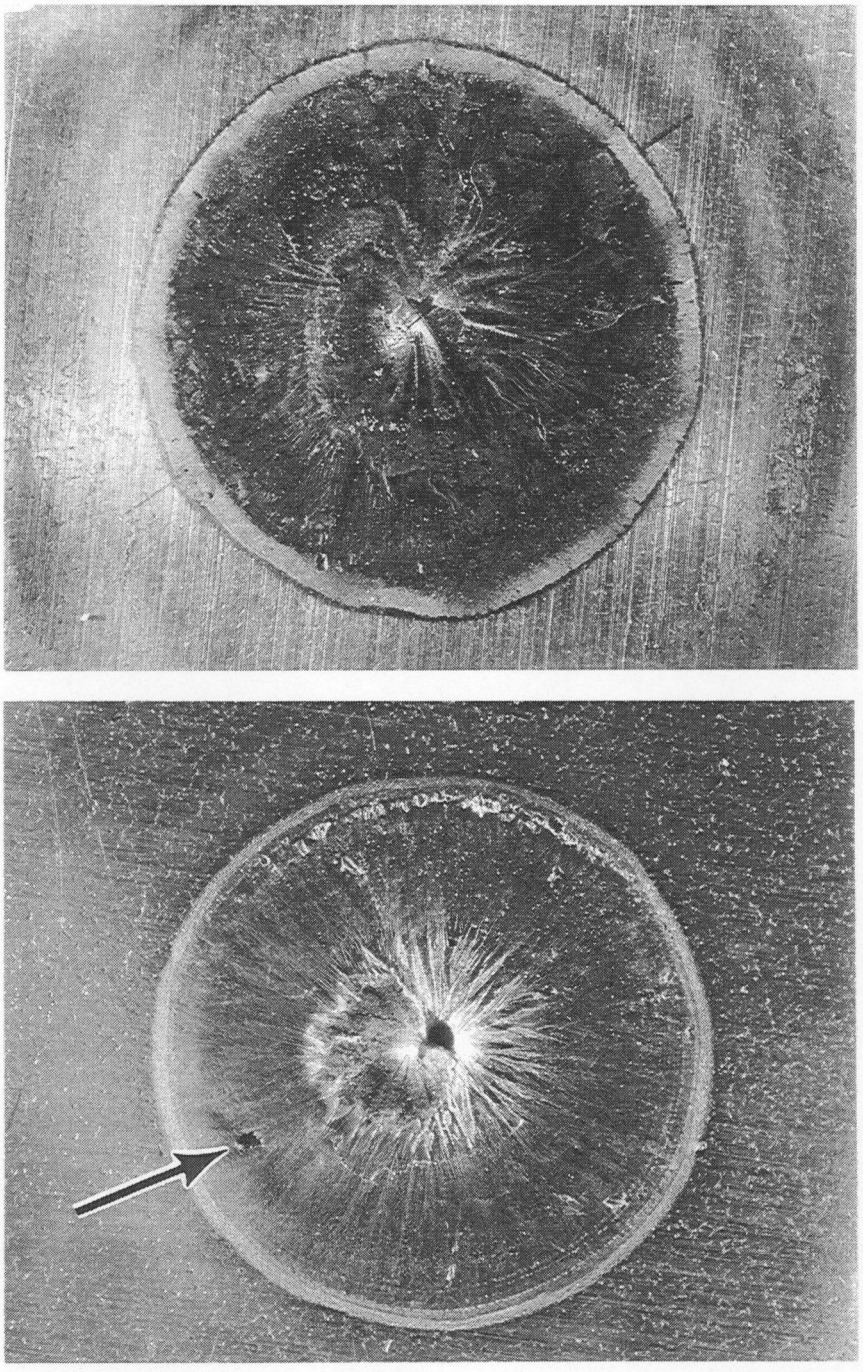
Temperature: 50^0C

Although ASTM practice G-48 requires the sample to be ground to a 120 grit finish prior to immersion in the test solution, a deviation was made for one set of coupons by retaining the surface in the as-welded state. This modification was deemed necessary because grinding erases the oxide differences in the samples welded with and without gas purification. Moreover, from the practical standpoint, the testing of as-welded surfaces has a more relevant result as opposed to using a 120 grit ground surface, wherein the oxide laden surfaces are erased.

6.9.3.1.4. **As-Welded Fusion Zone**

The samples were photo-documented prior to testing and later evaluated using stereo microscopy. Figures5 8 (a&b) and 59 (a&b) show the appearances of the spot welds made with and without gas purification, after immersion in a 6% $FeCl_3$ solution at 50^0C for 48 hours for conducting the pitting corrosion test. The approximate calculated contaminant levels originally blended into the gas were 300 ppm moisture and 100 ppm oxygen.

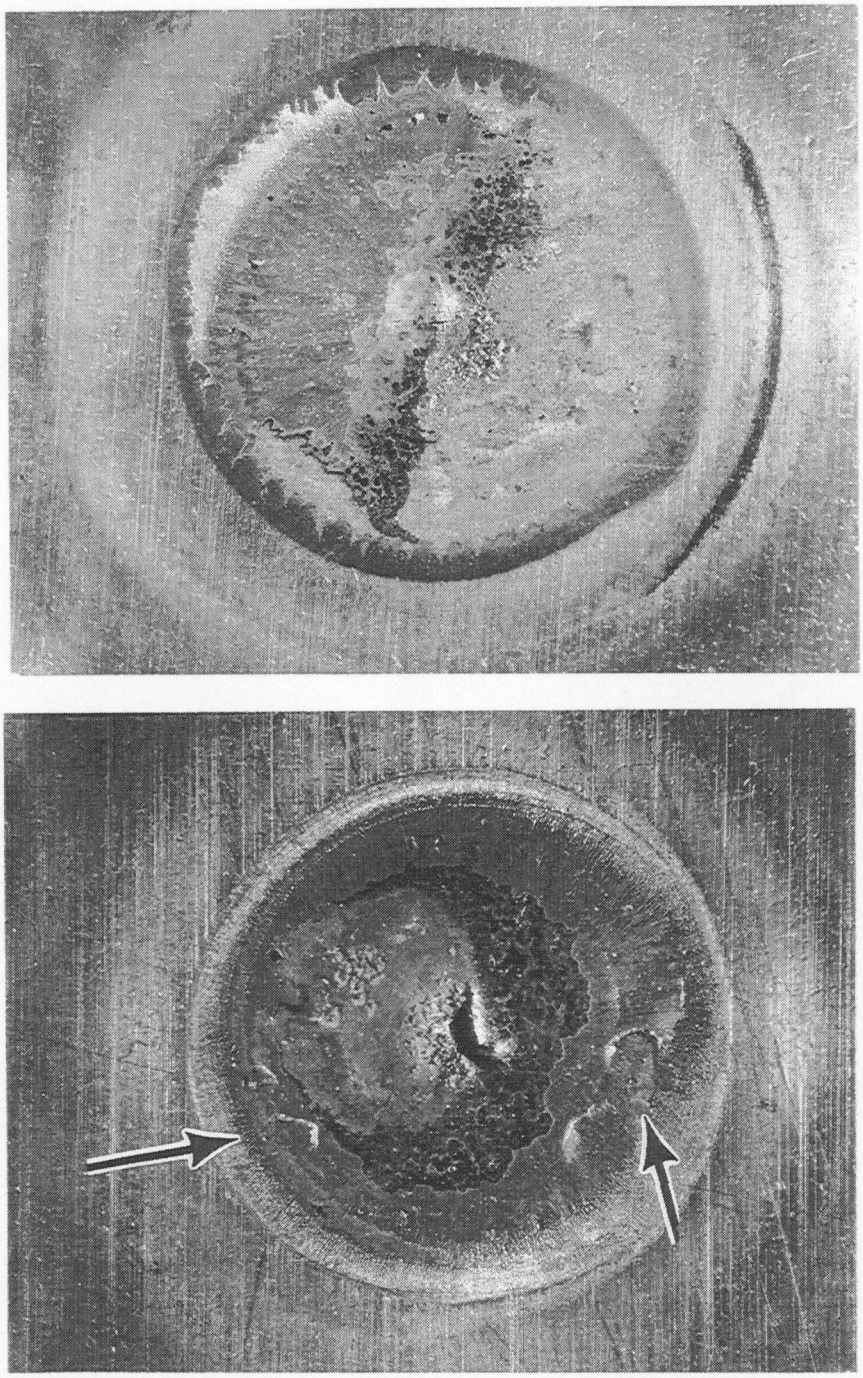
Figures 58a and 59a depict the appearance of the face surface, while figures 58b and 59b depict the root surface of the samples described above after the pitting corrosion test. Clear evidence of macropitting was observed on all surfaces. In addition, several other differences were also noticed. The heat tint colors on the sample welded without gas purification were completely erased on the face side of the coupon. Likewise, the light brown heat tint on the



(a)

(b)

Figure 58. Appearance of GTA spot weld on AL-6XN alloy shielded with purified argon gas after immersion corrosion testing in 6% $FeCl_3$ solution indicating macropitting. (a) face surface (b) root surface



(a)

(b)

Figure 59. Appearance of GTA spot weld on AL-6XN alloy shielded with oxygen and moisture contaminated argon gas after immersion corrosion testing in 6% $FeCl_3$ solution indicating macropitting. (a) face surface (b) root surface

face of the sample welded with gas purification was accentuated by corrosive attack. Evidently, the chromium-depleted region associated with the heat-tinted oxide was aggressively attacked. Corrosion debris was clearly evident on the face and root surfaces of the sample welded with the unpurified gas. Corrosive attack in the base metal within the gas coverage zone was noted, as seen in the crescent shaped band in figure 59a. The root, however, was severely attacked and was observed to have formed crater-like macro pits clearly visible across the fusion zone area.

Microsegregation in GTA autogenous welds of AL-6XN is a common problem and may have contributed to the localized corrosion evidenced only in the weld metal and not in the base metal.

6.9.3.1.5. **Fusion Zone (Ground)**

Two samples were also tested according to the exact ASTM practice G-48 recommendations. The samples were surface ground to 120 grit finish as opposed to testing them in the as-welded condition. In this case described above where the surfaces tested were in the as-welded condition, both samples experienced pitting. Both samples had a crater-like hole at the exact weld center, and it is around this crater that most of the pitting occurred. The pitting in the samples welded with and without gas purification are depicted in figures 60a and 60b. Micro segregation/coring normally occurs in austenitic weld metal dendrites. Localized regions within a weld can contain significantly less molybdenum than adjacent regions. Such molybdenum-depleted regions are much more susceptible to localized attack, even in moderately oxidizing

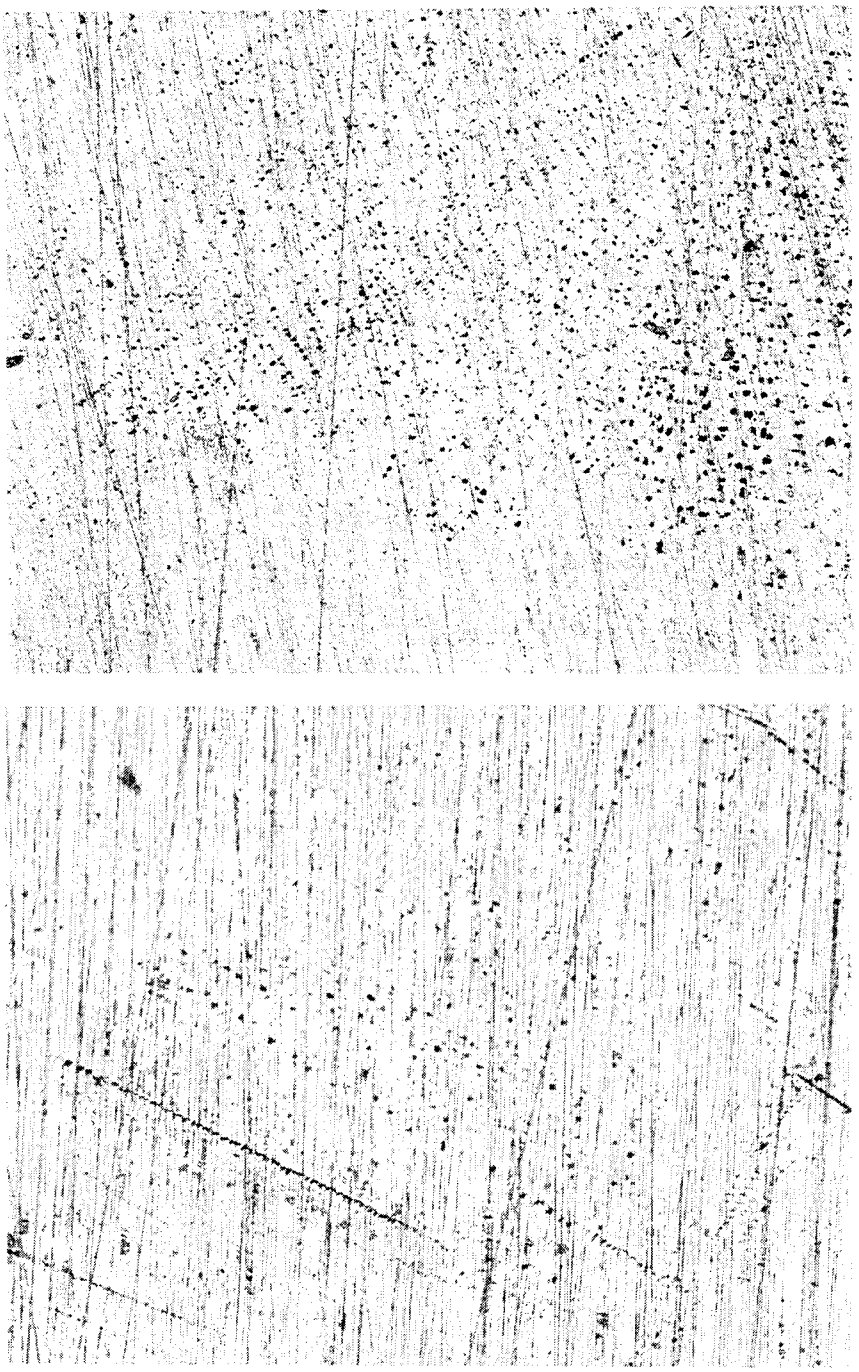


Figure 60. Appearance of ground fusion zone of GTA spot weld on AL-6XN alloy after immersion corrosion testing in 6% $FeCl_3$ solution indicating micropitting. (a) Weld shielded with Nanochem purified argon gas (b) Weld shielded with moisture and oxygen contaminated argon gas

solutions. Such attack is also most likely to occur in autogenous GTA welds. The probability of oxygen and hydrogen concentration in the crater is also high considering this is the last portion to solidify. Moreover, the columnar dendritic solidification substructure at the weld center is different from the otherwise cellular dendritic substructure that prevails in the weld region. All the reasons stated may have influenced preferential corrosion at the weld crater.

6.9.3.2. **Sodium Hydroxide Electrochemical Tests**

The data generated in the polarization tests in the experiments using 10% sulphuric acid and 6% ferric chloride generated data on the behavior of welds in aggressive acidic media. To complement this data, polarization tests were then conducted in 50% sodium hydroxide solution. In order to simulate behavior in a boiling caustic scrubber, it was decided to heat the test medium to boiling. The results of the tests are indicated in figure 61. This medium proved to be extremely aggressive as indicated by the negative free corrosion potentials. Also, the onset of corrosion at a relatively earlier potential (550 mV for the sample welded with purified gas), and 100 mV for the sample welded with contaminated gas, indicate that corrosion resistance is lowered in the boiling caustic solution as compared to the ambient temperature sulphuric acid or ferric chloride test medium. Comparison of the corrosion behaviors of the two samples also yields interesting results. The E_{corr} values differ by 350 mV, with the sample welded with purified gas being the more noble of the two. Likewise, the corrosion current density at the free corrosion potential is higher by more than two orders of magnitude for the sample welded with contaminated gas,

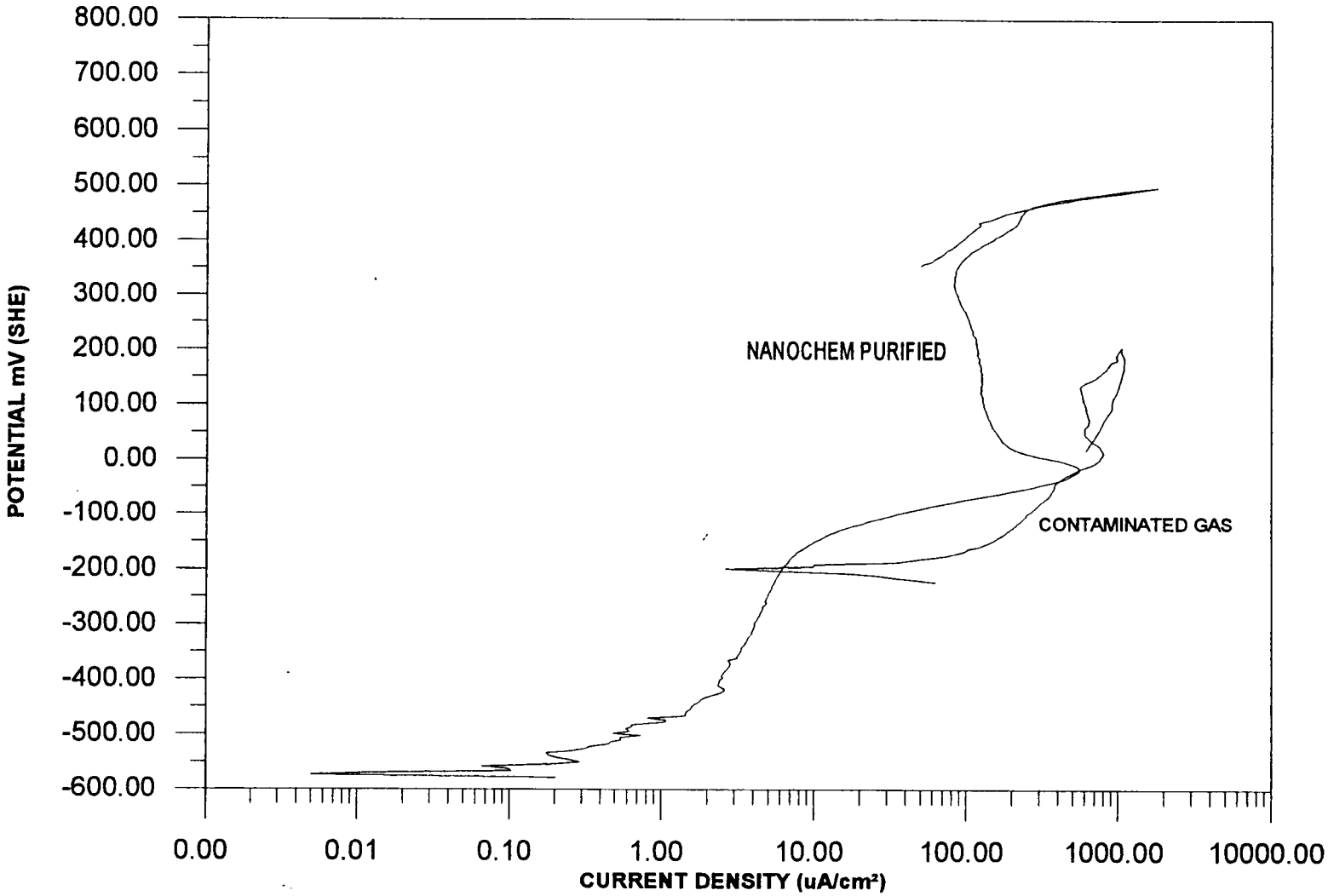


Figure 61. Anodic polarization behavior of GTA, as-welded AL-6XN alloy shielded with contaminated and purified argon gas, in boiling 50% NaOH solution

indicating a poor corrosion resistance to start with. Also, the extent of passivation in the sample welded with contaminated gas is comparatively very limited. Passivation is observed for only 100 mV before the onset of active corrosion associated with an anodic current density of 1000 uA/cm^2, which in this case was the set threshold current density. However, pits initiated in the sample exhibited a tendency to repassivate.

Moreover, the reverse scan generates hysterisis only in the sample welded with contaminated gas, indicating minor pitting. However, the current traces back along the up-scan curve for the sample welded with pure gas, indicating the total absence of pitting.

The test data for the sample welded with impure gas had to be attempted unsuccessfully on three occasions because of damage to the reference electrode tip (heat shrink tubing expands and causese the porous filter tip to fall away). Following this test, no further tests were conducted in this medium for risk posed to the corrosion test-cell equipment. (The use of a Luggen probe, however, may offer a means to overcome this experimental constraint .)

6.9.3.3. **Sulphuric Acid Electrochemical Tests**

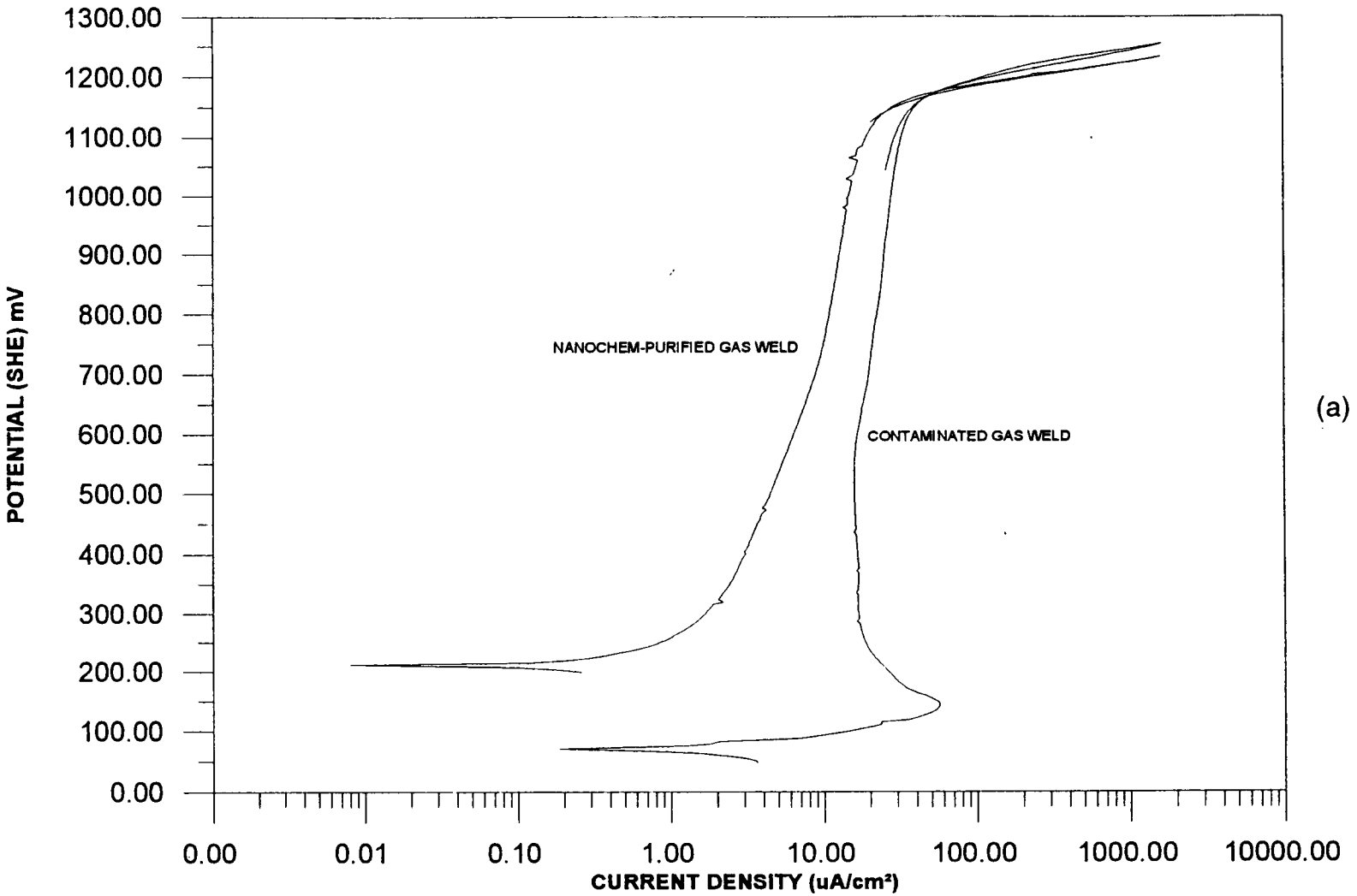
Extensive polarization tests were carried out in 10% sulphuric acid test solution. Not only is this medium industrially relevant, but is also one of the most aggressive mediums for testing AL-6XN alloys.

6.9.3.3.1. **Heat Tint Area**

As described in the previous sections with relevant pictures, the welds made with contaminated gas were characterized by extensive heat tinting. No heat tint colors were detected in the samples welded with purified gas. As alluded to earlier, such heat tints are deleterious to the corrosion resistance of the stainless alloys. It was decided to examine the exact difference in corrosion behavior by performing anodic pitting polarization test scans. Accordingly, the samples were prepared by masking off the entire coupon area barring the heat tinted region. The results of the polarization scans conducted on the heat tinted areas are illustrated in figure 62a. A cursory inspection reveals that the sample welded with contaminated gas and bearing the heat tinted area is associated with a free corrosion potential that is less noble, and a passive current density that is higher than the sample welded with purified gas. Qualitatively, this observation implies a lower corrosion resistance. No pitting, however, was observed in both samples. The difference between the two samples does not, however, translate into substantially different service performance. The difference can be treated as more qualitative than quantitative.

6.9.3.3.2. **As-Welded Surface**

Polarization scans of the as-welded surfaces of the two samples welded with and without purification are presented in figure 62b. The most striking feature is the difference in the corrosion current densities of the two samples in the passive range. The sample welded with purified gas starts out at a relatively



(a)

Figure 62. Effect of shield gas purity on the anodic polarization behavior of GTA spot welded AL-6XN alloy in 10% H_2SO_4. (a) Heat tinted region

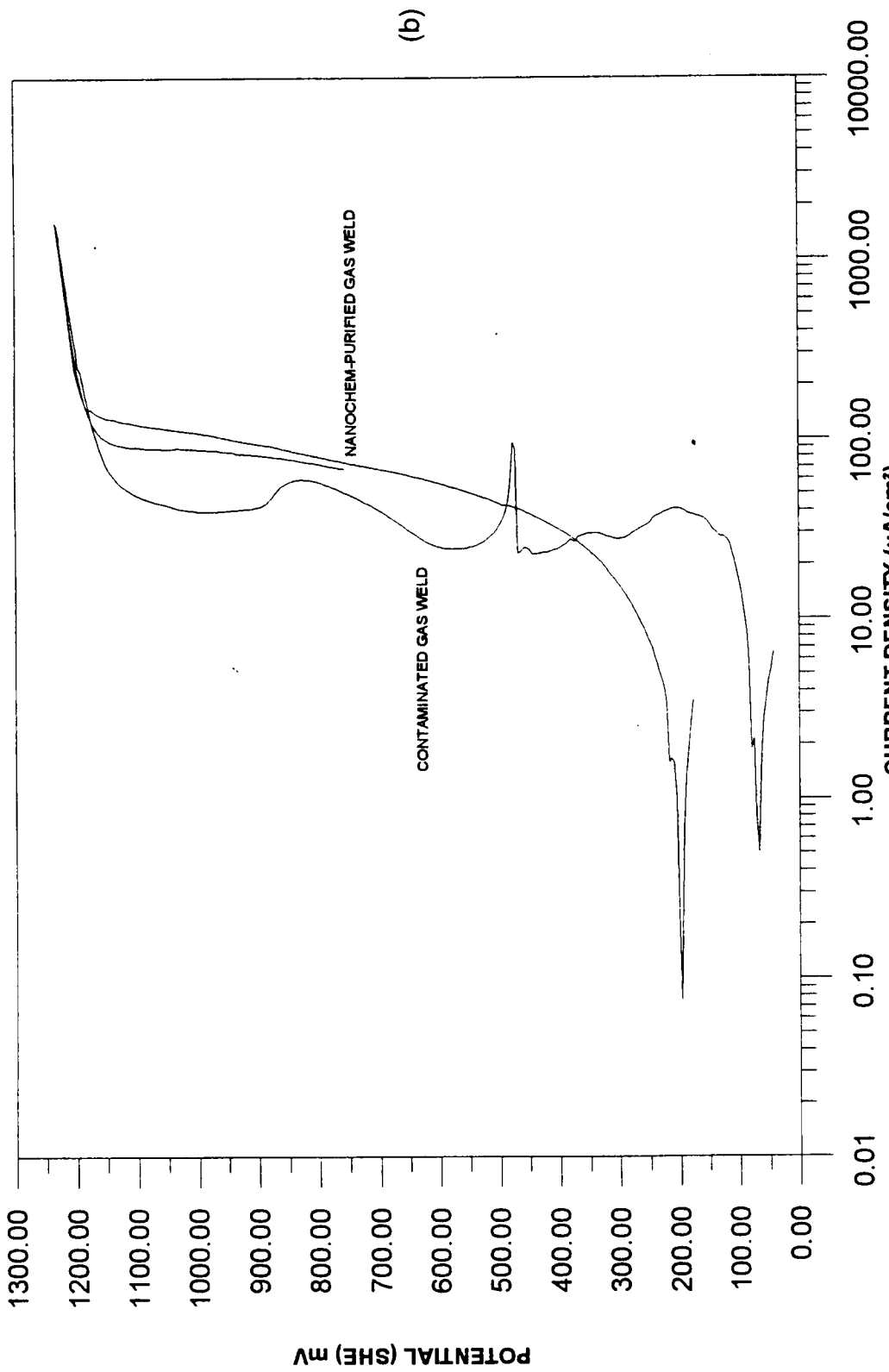
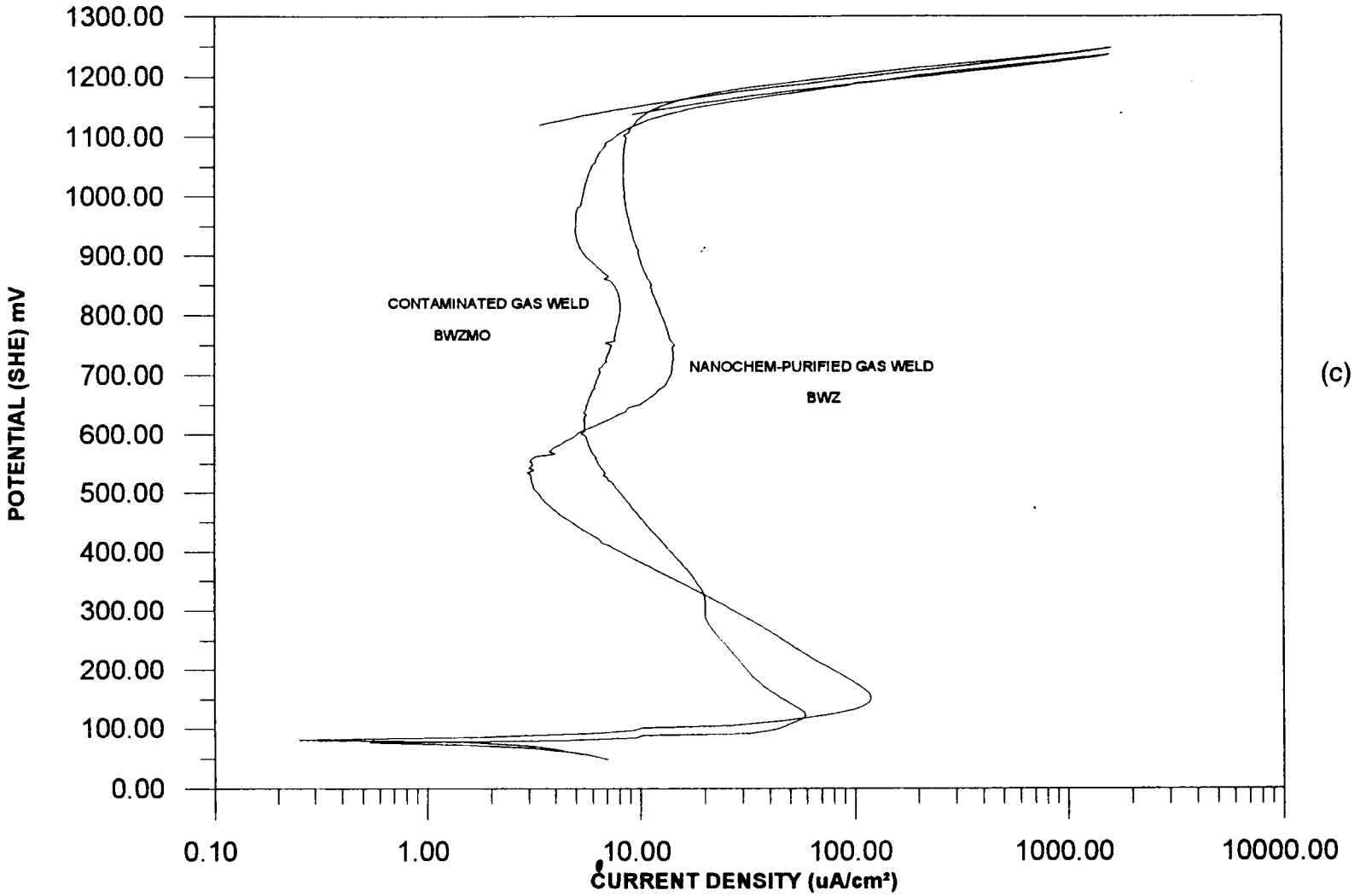


Figure 62 (continued). (b) as-welded fusion zone region



(c)

Figure 62 (continued). (c) Entire weldment

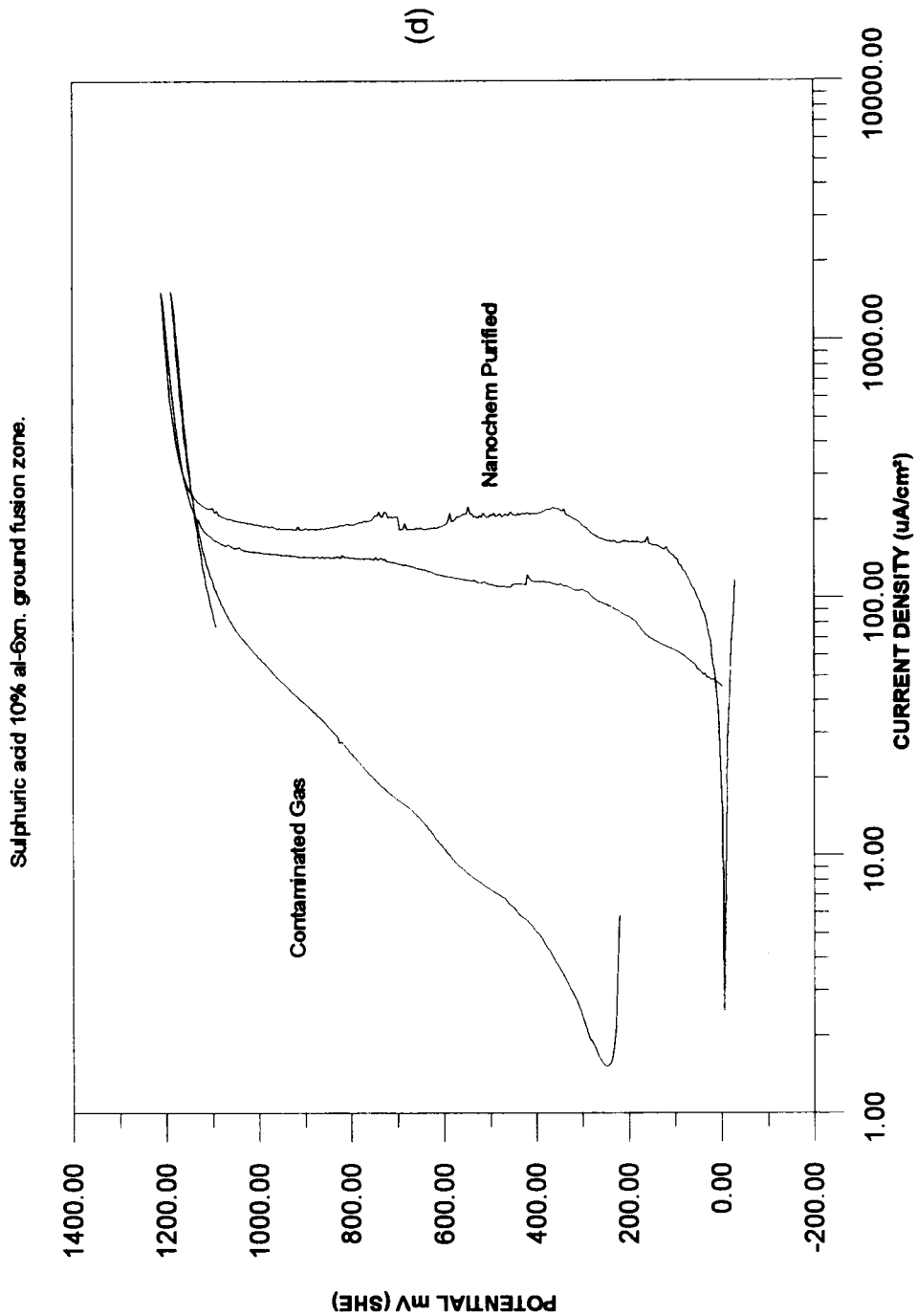


Figure 62 (continued). (d) Ground fusion zone region

more noble potential (200 mV as opposed to 50 mV) and then steadily passivates at around 80uA/cm2 current density. On the other hand, the anodic current density of the sample welded without purification fluctuates significantly during passivation. Fluctuation even varies as much as between 20 to 100 uA/cm^2 at around 450 mV. This fluctuation can be attributed to the thick, non-uniform, debris-covered surface of the specimen pictorially illustrated in previous sections. By the same token, the existing oxide accounts for the lower overall passive current density compared to the higher passive current density associated with the fresh passive film formed on the shiny, clean, oxide-free surface of the sample welded with purified gas.

6.9.3.3.3. **Weldment (HAZ, FZ, and base metal)**

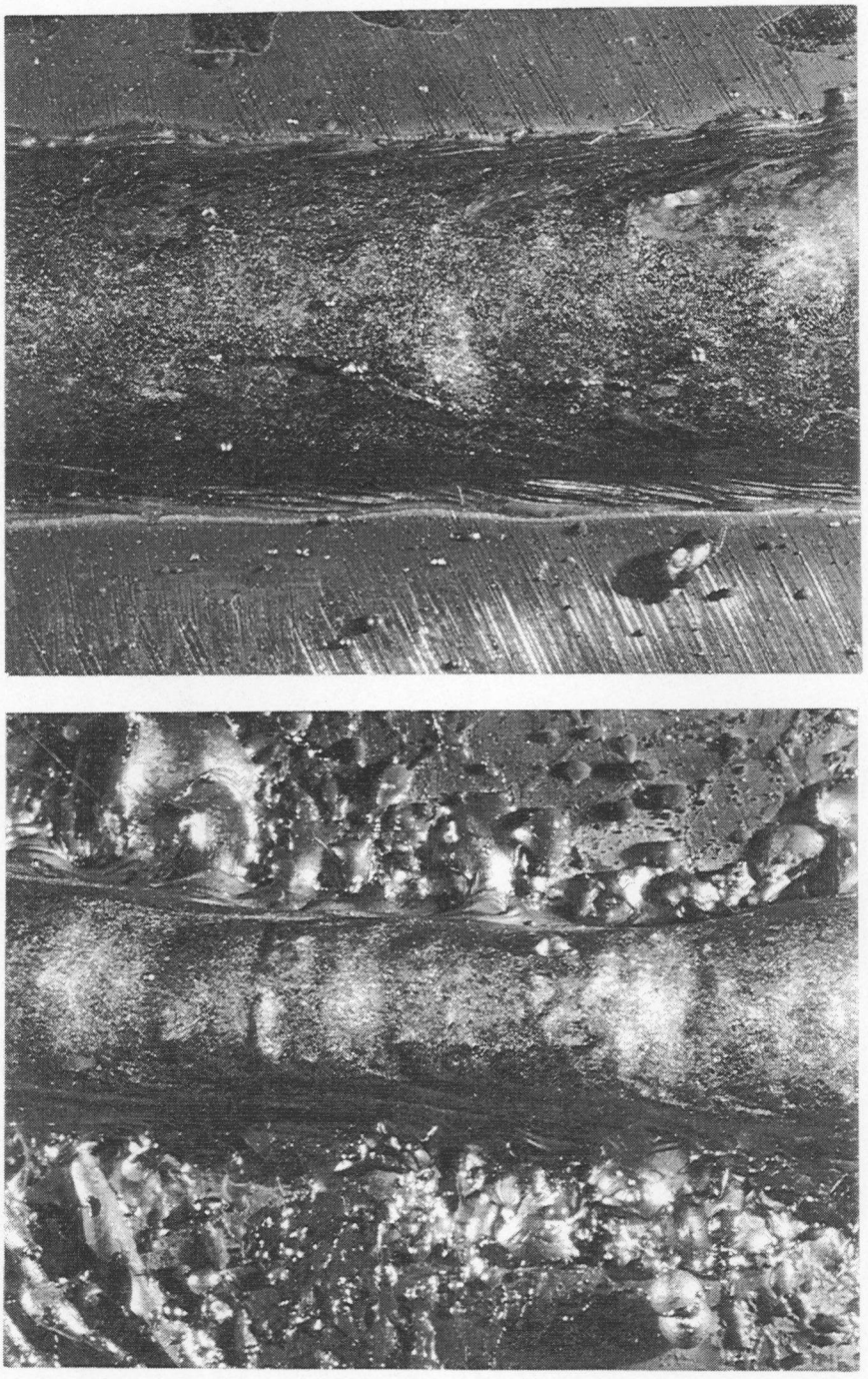
Polarization data for samples welded with and without purification is presented in figure 62c. The free corrosion potentials are identical, and overall, the two scans are quite similar. The only fleeting difference is that the sample welded with impure gas starts to passivate earlier than the sample welded with purified gas and is associated with an anodic current density that is lower than that of the sample welded with pure gas. This difference stems from the existing oxide/passive film on the sample welded with impure gas.

6.9.3.3.4. **Fusion Zone (ground)**

The polarization scans generated by testing the fusion zones of the samples welded with and without purification of the shield gas after they had been ground to 600 grit are presented in figure 62d. Passivation in the sample welded with pure gas initiates at 100 uA/cm2 and remains stable until 1200 mV. Beyond that potential, there is a rapid rise in anodic current density, probably caused by oxygen evolution due to instability and breakdown of water. On down scanning from the threshold current density of 1000 uA/cm2, no hysterisis is created, confirming that no pitting has taken place and that the rise in anodic current density is only due to oxygen evolution.

6.10. **100-S Filler Wire: GMA Weld Tests**

The purpose of the preliminary two GMA weld tests with 100-S filler wire was to ascertain whether deviating from the recommended use of active gas component (2% oxygen) in the shield gas would have any effect on the welding characteristics. Figure 63a indicates a weld made with the recommended argon-2%oxygen. The weld was deposited using conventional GMA techniques. The arc was stable and the resulting weld bead is fully acceptable in terms of appearance and absence of weld defects. However, in the second weld shielded with pure argon and welded at the same parameters, the weld and arc characteristics were totally degraded. Evidence of this degradation is seen in figure 63b. Almost 50% of the melted 100-S filler wire was lost as spatter. Evidently, the presence of the recommended active component (2%



(a)

(b)

Figure 63. Surface appearance of GMA weld deposit of 100-S filler wire indicating the necessity of an active component in the shield gas. (a) recommended Ar-2% oxygen shield (b) argon shield

oxygen, in this case) is essential for good arc characteristics and bead appearance. Violation of this prescription results in unsightly welds with poor operational characteristics. The beneficial effect of oxygen in improving arc characteristics and bead appearance has been alluded to in the introductory section.

Unfortunately, active shield gas additives such as oxygen and carbon dioxide pose problems when used in conjunction with Nanochem welding grade purifiers. Nanochem is chemically incompatible with active gases and hence cannot be used with shield gases that contain these additives. Use of gases containing active additives not only degrades the resin, but also poses a serious safety hazard. Evidently, the progress of the GMA weld tests in terms of conducting the proposed cold cracking tests and the mechanical property tests were jeopardized. It was at this point that attention was focused on GTA welding, which inherently prohibits the use of active shield gas additives, thereby allowing utilization of the Nanochem purification unit.

6.11. **100-S GTA Weld Tests**

The GTA weld tests were designed to examine the effects of shielding gas moisture in terms of the weld diffusible hydrogen levels produced, which in turn affects the cold cracking susceptibility of carbon steel welds.

The results of the diffusible hydrogen test measurements are depicted in figure 64. The greatest effect of contamination was seen in the coupon welded with the system gas line totally saturated with moisture. (Water was passed through the gas line for two seconds following which the line was drained until

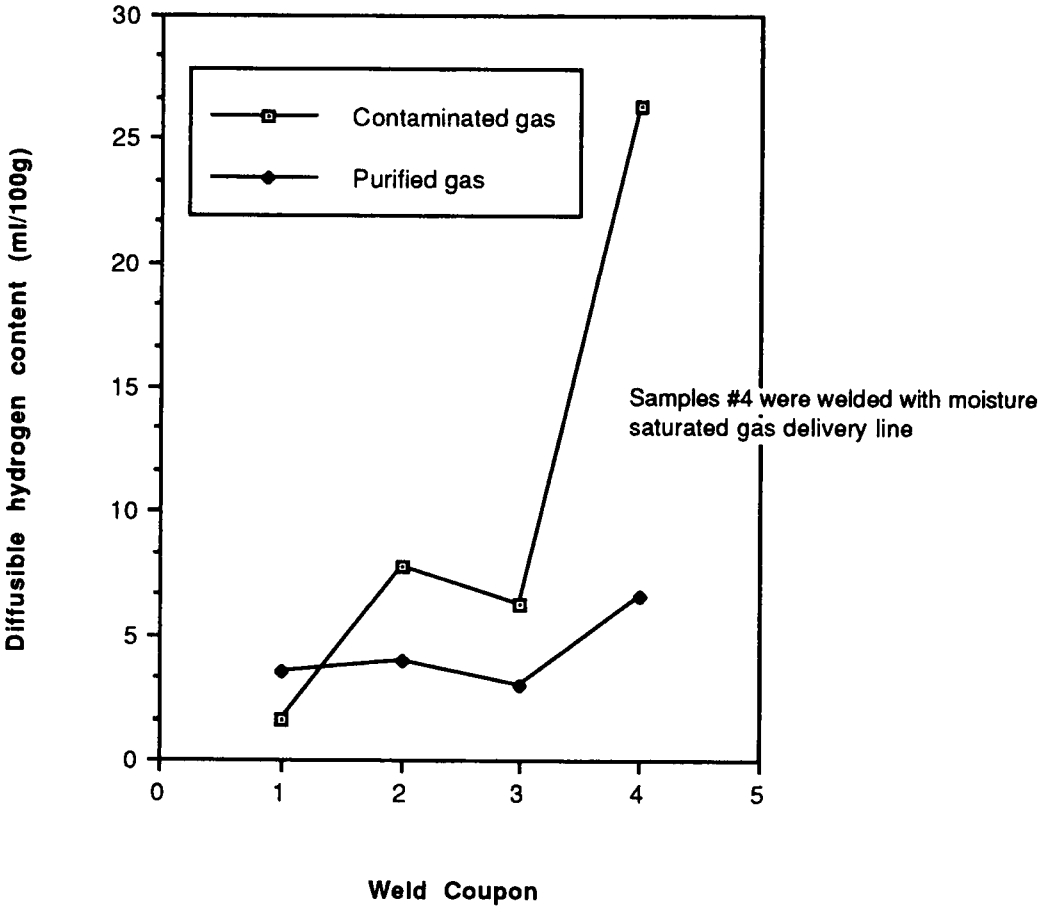


Figure 64. Diffusible hydrogen content in GTA weld deposits of 100-S filler wire, shielded with moisture contaminated and Nanochem-purified helium gas

no traces of water were visible on the translucent surface of the polypropylene tubing.) The diffusible hydrogen content was a high 26.3 ppm. In the same situation, the use of Nanochem purification was effective in dropping the level of weld metal diffusible hydrogen to a mere 6.6 ppm-a reduction of almost 20ppm hydrogen.

For the coupons where the moisture and oxygen contaminants were only added as an impurity in the shield stream, the relative difference in the diffusible hydrogen levels of the contaminated gas welds (5.2 ppm) and the Nanochem-purified welds (3.5 ppm) was not as pronounced. The coupon with the lowest diffusible hydrogen (1.6ppm/100 g) was interestingly also one of the samples welded with impure gas. This low reading is most probably due to experimental error because it is totally in contrast to the observed trend, where samples welded with the contaminated gas consistently exhibited higher diffusible hydrogen levels. Such experimental errors can arise due to delay in transferring the as-welded coupon from the weld fixture to the ice water, then to the liquid nitrogen bath, and finally into the sampler.

During welding, the arc initiation was observed to be difficult in the samples welded with contaminated gas . In fact, after the first sample was welded, the electrode had to be reground prior to welding each of the following three coupons. This was partly due to the fact that the oxygen added into the shield stream lead to partial oxidation of the electrode in the tip area, where the arc is initiated. Moreover, the presence of moisture and oxygen also alter the ionization characteristics of the gas, making it more difficult for the arc to be initiated. No visible defects, cracks, porosity, or excessive slag were seen in both sets of samples welded with and without purified gas.

A situation where the gas plumbing of a system could get saturated with moisture, such as that simulated above, is not unlikely in a field situation where improper storage of hoses, open exposure to rain, leaky fittings, etc. are sometimes common. Such situations lead to adsorbed moisture build-up within the gas lines, which is then subsequently released along with the shield gas into the weld pool during welding. In the case of HY steels, this results in high diffusible hydrogen levels as the tests conducted have shown. In concert with stress and a crack-susceptible, high hardness, martensitic microstructure, these high levels of diffusible hydrogen can easily induce cold/delayed cracking. Use of Nanochem clearly alleviates the situation.

In conclusion, the 20 ppm reduction of diffusible hydrogen observed in the test clearly proved that Nanochem can allieviate detrimental effects caused by defective gas plumbing and contamination. However, it must also be realized that moisture entering the system upstream of the Nanochem purifier cannot be prevented from causing its damage. Adequate precautions must therefore be taken to ensure gas line fitness for service.

6.12. **Filler-Added GTA Weld Tests: DSS 255**

Figure 65 depicts a filler-added GTA weld made on 255 duplex stainless steel. The effects of moisture and oxygen contamination in the shield gas are clearly evident in the weld stop. Compared to the autogenous welds made on 304 stainless steel, DSS 255 steel appeared to be more forgiving to the presence of contaminants in the shield gas. Unlike the 304 stainless steel welds that were degraded along the entire length of the weld, the 255 weld

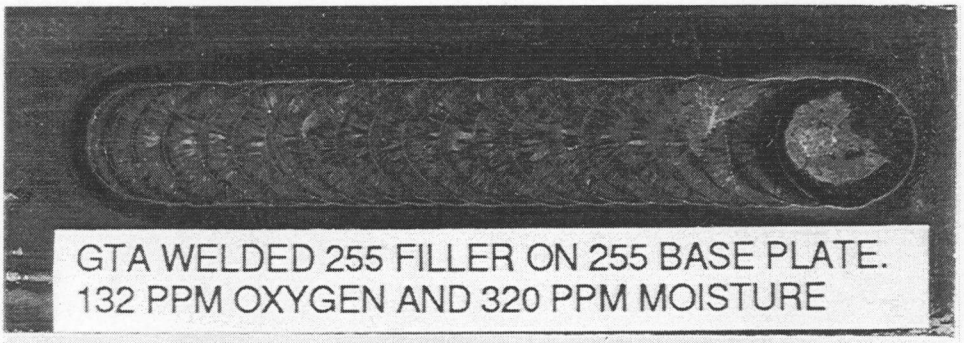


Figure 65. GTA filler-added weld in 255 duplex stainless steel shielded with moisture and oxygen contaminated gas showing weld crater contamination

remained oxide-free up to the near crater and crater area. In fact, as evidenced in figure 65, even the solidification substructure is clearly visible along the length of the weld. Apparently, the contaminants are swept along with the arc up to the crater area, where the arc is extinguished and the oxide contaminants are rejected from the weld pool to solidify on the surface.

When only oxygen was used as the shield gas contaminant, the results were similar to that seen in 304 stainless steel. The degree of oxidation was relatively less as compared to when both moisture and oxygen were added to the shield gas. One set of autogenous welds was also made with the moisture- and oxygen-contaminated argon gas purified with Nanochem before supply to the torch. The most significant indicator that purification had been successfully effected was the appearance of the weld stop area. In contrast to the weld made with contaminated gas, the crater of the weld made with repurified gas was shiny and devoid of any colorations. However, a small oxide island was noted in each of the repurified gas welds at the exact point below the electrode tip.

The three sets of welds described in the above section were not made using identical welding parameters and welding modes (filler added vs. autogenous). Hence, no direct comparisons are drawn between the welds. However, the effect of moisture and oxygen impurities in causing a heavy blanket of oxide at the weld crater is firmly established.

6.13. **GMA 2205 Weld Tests**

6.13.1. **Arc Characteristics**

The arc characteristics in terms of metal transfer were the most optimum in the assembly shielded with argon-2% oxygen. Spray transfer mode was easily achieved using this gas at the parameters used. In contrast, the transfer mode when welding with argon shield devoid of any oxygen component was mostly short arc or globular and inherently associated with more spatter. This feature, however, may or may not be a disadvantage when welding in the field. In overhead and vertical positions, where spray transfer is practically useless, it is this type of a globular, short-circuiting transfer which is mostly preferred.

The oxygen component of the shield gas, on the other hand, increases the working envelope of the welding parameters, thus providing a wider range of characteristics. No evidence was found in the literature indicating use of pure argon shielding when GMA welding the duplex stainless steels. The most popular method is to combine an active component such as oxygen or CO_2 with the main inert argon or argon-helium shield.

6.13.2. **Bend Testing**

All assemblies were bend tested as per ASME code procedures. All assemblies, except the assembly welded with welding grade argon, passed the test. No defects exceeding 3 mm were found on the convex, tension surfaces of the bend samples. Some minor cracks did originate in the sample welded with

moisture-contaminated argon at the specimen corner but did not propagte to 3 mm and, hence, was not relevant according to the ASME specifications followed.

6.13.3. **Corrosion Behavior**

All weld samples were electrochemically tested in accordance with ASTM G-48 procedures (6% ferric chloride solution, ground surface). The anodic polarization scans of the samples welded with the four, varied purity argon shields are indicated in figure 66. As visible in the scans, no noteworthy differences were found between any of the samples. The free corroding potentials differed by a maximum of 75 mV. The passive current densities differed by a maximum of 60 uA/cm^2. However, a significant difference was found between the samples welded with the four argon shields and the sample welded with argon-2% oxygen shield. As seen in figure 67, the sample welded with argon-2% oxygen shield is significantly different compared to the samples welded with the four argon shields of varied purity. The passivation current density indicates that there is noticeable localized pitting, immidiately followed by repassivation of the pits. The current density associated with this activity is less than that of the samples shielded with fully argon gas. Finally, after repassivation of all initiated local pits, a uniform corrosion mechanism comes into effect. The potential at which the passivity changes to uniform corrosion is the same potential at which passivity is lost in the four samples welded with the varied purity argon shields. The sample shielded with the argon-2%oxygen, however, does not rapidly corrode thereafter. Instead, the current density rises

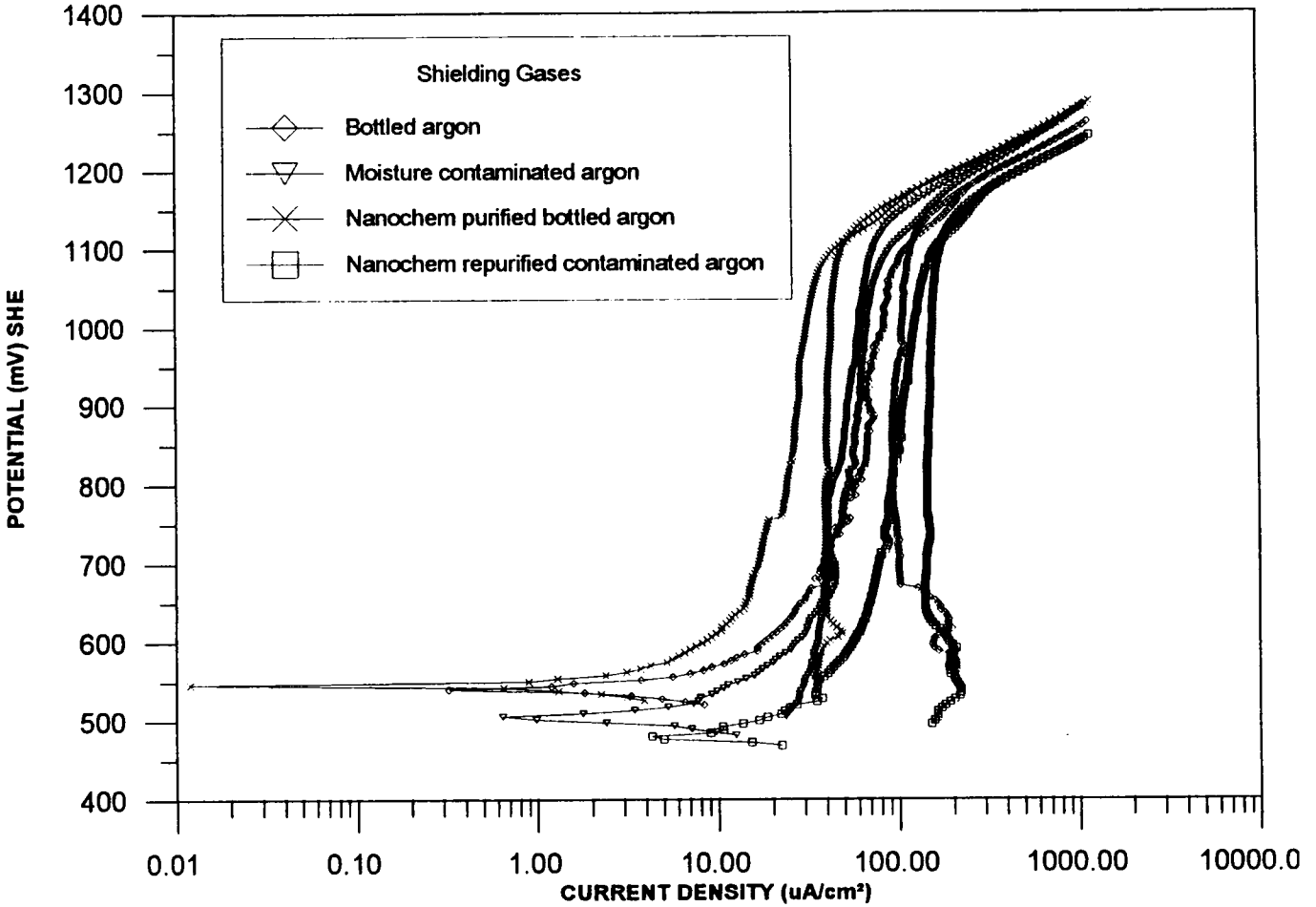


Figure 66. Electrochemical anodic polarization behavior of alloy 2205 GMA weldments, shielded with varied purity argon gas, in 6% ferric chloride solution.

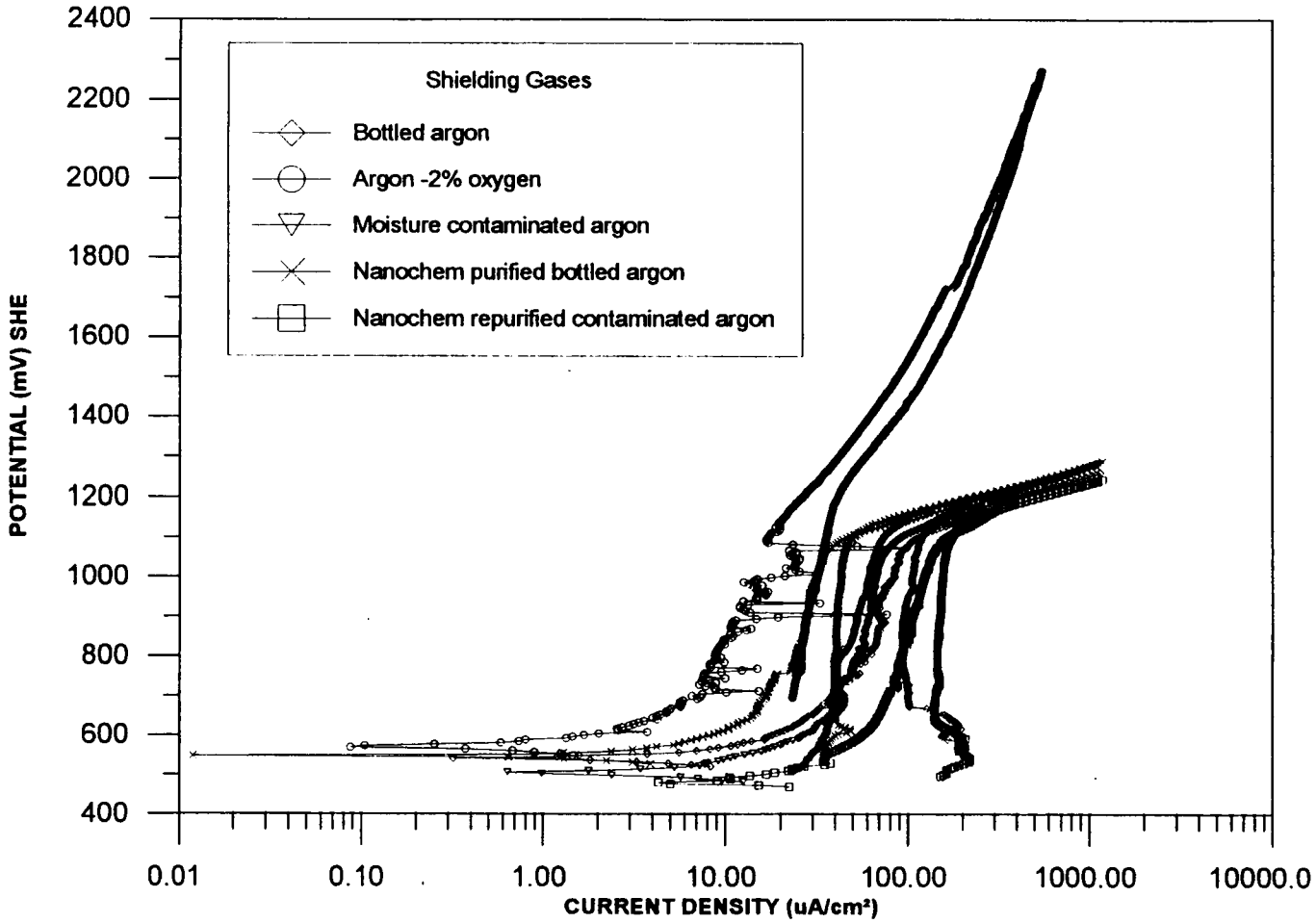


Figure 67. Electrochemical polarization behavior of alloy 2205 GMA weldments illustrating the difference produced by argon -2%oxygen shield and varied purity argon shields

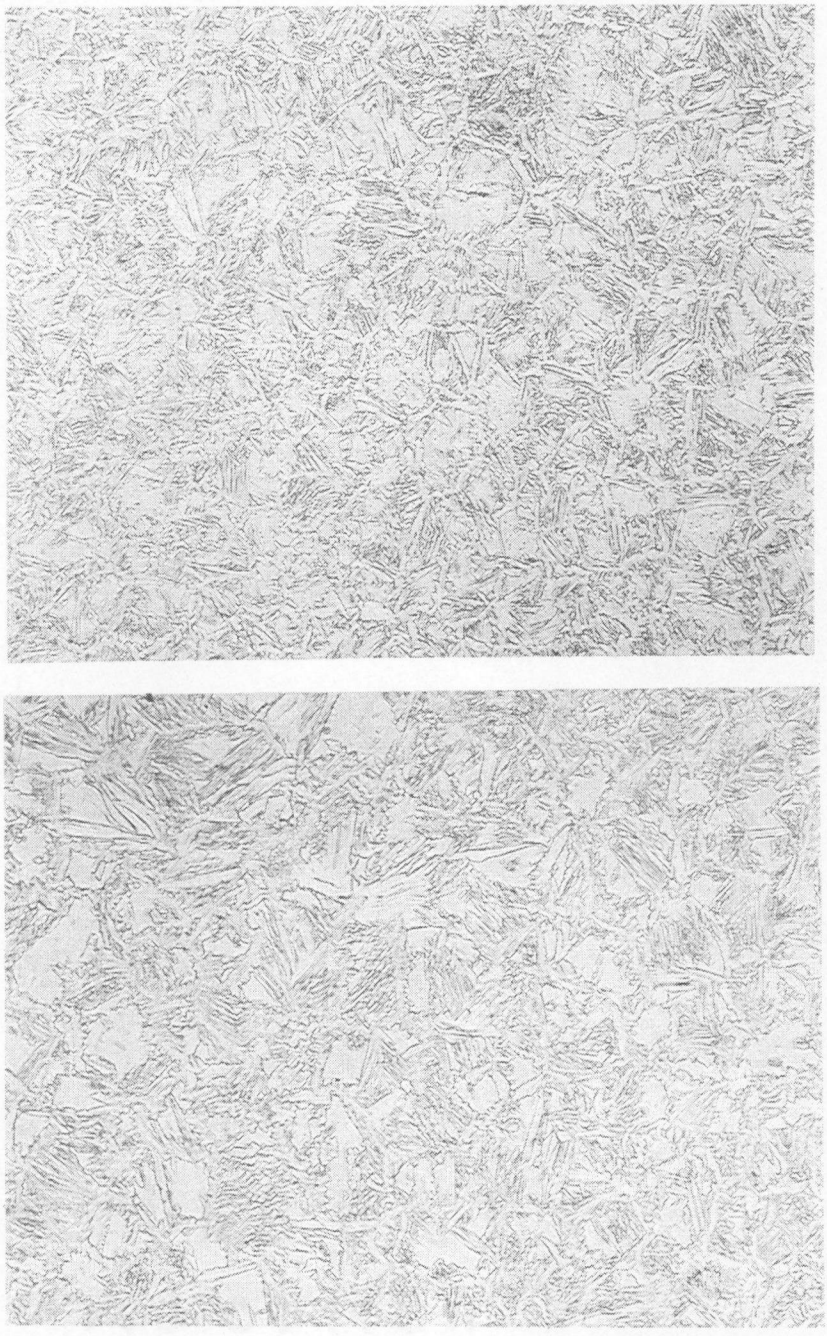
so slowly that the threshold current density (1000 uA/cm^2) is not reached even 1000 mV after the passive film breakdown potential.

The behavior described seems to suggest that the argon-2%oxygen shielded weld is qualitatively more corrosion resistant than the welds shielded with argon devoid of the oxygen additive.

6.13.4. **Microstructure**

All samples were examined after corrosion testing. The corrosion effect was clearly manifested in the strong etching effect created on the 600 grit polished samples. The etched samples were evaluated for microstructural characteristics.

No microstructural differences were found between the samples welded with the four varied purity argon shields. Figure 68a, 68b, 68c, and 68d, depict the appearances of the samples shielded with welding grade argon, argon contaminated with moisture, Nanochem-purified welding grade argon, and Nanochem-purified argon originally contaminated with 300 ppm moisture. The typical microstructure of all the four samples described is the same. The ferrite grains (dark regions) are surrounded by austenite that has an acicular structure. In contrast to this typical appearance, the sample welded with argon-2%oxygen, showed a characteristically significantly different microstructure. The microstructure of this sample is depicted in figure 69a (For comparison, the weld shielded with Nanochem-purified argon is also depicted in figure 69b.) Evidently, the presence of oxygen influences the microstructure, by altering the transformation kinetics and also rendering the final microstructure more coarse.



(a)

(b)

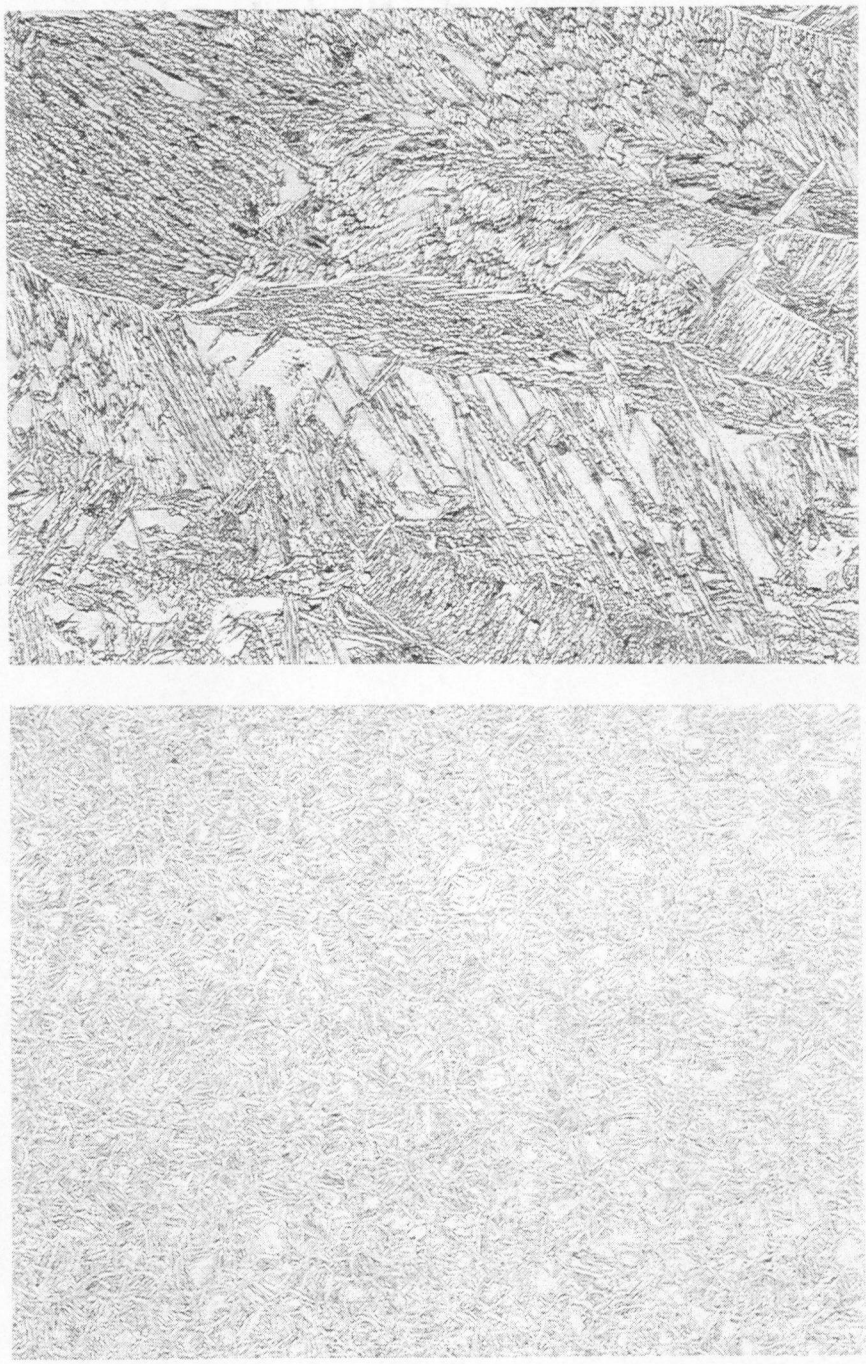
Figure 68. Microstructure of GMA welded 2205 duplex stainless steel.
(a) welding grade argon shield
(b) argon shield contaminated with approximately 300 ppm moisture



(c)

(d)

Figure 68 (continued). (c) Nanochem-purified, welding grade argon shield (d) Nanochem-purified argon shield originally contaminated with moisture



(a)

(b)

Figure 69. Microstructure of GMA welded alloy 2205 duplex stainless steel.
(a) industry recommended argon-2%oxygen shield
(b) Nanochem-purified argon shield

All the welded assemblies also indicated some porosity and lack-of-fusion defects. In the weld shielded with argon-2% oxygen, lack-of-fusion defects were found. This feature is an already known limitation of welding with argon-2% oxygen shields. Whether or not the porosity defects found in the welds made with the argon shield was inherent in the process itself was not established. The amount of defects seen was not significant and did not affect any of the tests conducted.

6.13.5. **Impact Toughness**

A total of five charpy impact samples were tested from each of the assemblies welded. Results are displayed in figure 70. The weld shielded with Nanochem-purified gas (originally contaminated with moisture) ranked the best in terms of impact toughness properties. In contrast, the weld shielded with the recommended blend of argon -2%oxygen exhibited the lowest toughness properties. The microstructure studied in the previous section offers an explanation for the impact behavior exhibited. The fine grain structure of the weld metal in the welds shielded with argon gas devoid of any oxygen component, as opposed to the coarser structure and inlcusion level of the weld made with argon-2%oxygen gas, is responsible for the improvement in toughness. The alloy losses caused by the 2% oxygen in the shielding gas is also largely responsible for the degradation in impact properties.

In spite of the loss in toughness, the popular gas for welding the duplex stainless steels still continues to be argon-2%oxygen. Evidently, the importance of arc characteristics is accorded more importance than toughness

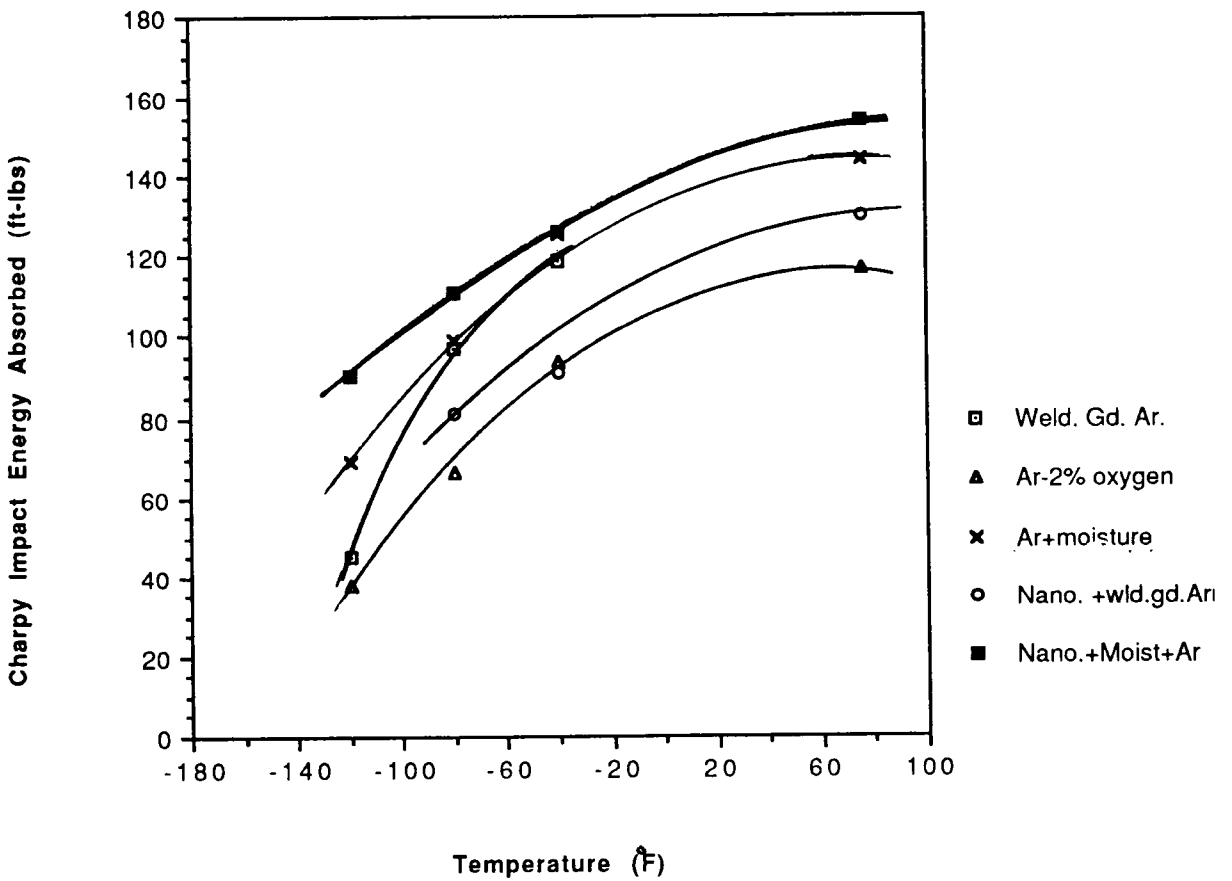


Figure 70. Impact toughness properties of GMA welded 2205 duplex stainless steel, shielded with different argon-based gases of varied purity

properties. The results generated in this test should offer a strong motivation for rethinking the existing shielding gas selection rationale.

The effects of moisture in the argon shield gas did not seem to be a significant deterrent to the impact properties. This fact is clearly visible from the relative closeness of impact toughness values of welds shielded with contaminated and repurified gas. Another feature observed is that impact values seem to reach a plateau at room temperature, which in this study was the highest test temperature. At the lower extreme of temperature (-120^{0}F), none of the samples exhibited zero toughness, owing to the austenite present in the weld metal. Although the toughness at -120^{0}F for the sample welded with welding grade argon, and the toughness at 75^{0}F for the sample welded with Nanochem-purified welding grade argon could not be determined for a lack of test samples, the trend is nevertheless well established. The weld shielded with Nanochem-purified welding grade argon ranks second lowest in toughness properties. Although this weldment passed the ASME bend test, evident lack-of-fusion defects were observed when machining the assembly for sample extraction. This finding suggests that the low toughness behavior may have been hampered by the defects generated in the welding process.

6.14. **Gas Injection Results: GMAW Torch**

The necessity and effectiveness of gas injection in the GA-50A torch, when modified in accordance with the patent description, were found to be debatable. The problems associated with the torch modifications, the solutions

devised, the concepts involved in the design changes made, and the observations recorded are presented below.

First, it was observed that a positive gas flow and pressure already exists at the cable inlet end and the contact tip end. When using argon shielding gas, gas flow rates up to 1.2 cfh at 0.042 psi were recorded at the cable inlet/adapter end of the torch cable. (The cable inlet/adapter end of the torch cable is the area where air entrainment, if any, is expected to occur). When using helium shielding gas fo rshielding, exit gas flow rates greater than 1.6 cfh were recorded at the cable inlet end of the torch cable. This finding negated the premise that owing to negative gas pressure and flow at the wire inlet and the contact tip ends, air entrainment could occur in this GMA torch. On the contrary, the positive pressures and gas flows, although small, are capable of preventing air ingress.

The presence of positive pressures at the two ends of the wire cable is easy to explain. The regular shielding gas flowing around the liner enters the liner easily because of its monocoil construction. This gas automatically pressurizes the liner throughout its length. Therefore, attempting to inject gas into this type of mono-coil liner is redundant. In fact, the liner itself could be looked upon as having injection points all along its length, owing to its monocoil construction.

In spite of the facts described above, the injection modification was still implemented in an attempt to examine whether the already existing positive gas flows could be further enhanced. Measurements revealed that although the flow rates at the contact tip end could be increased by the modification, it did not serve to increase any of the existing flow rates and flow pressures at the cable

inlet end- the most susceptible area for air ingress into the liner. On the other hand, it was found that simply increasing the regular shielding gas flow rate increases the flow rates at the cable inlet end. This finding proves that the regular shield gas does exit the torch cable at the wire inlet end, thus automatically generating a positive pressure that prevents the entrainment of air.

Moreover, the difficulty of injecting gas into the liner near the contact tip end and expecting it to exit efficiently through the cable inlet end is easily explained. One primary reason is the large distance at which the gas injector is located from the cable inlet end (7 feet). This difficulty is compounded by the high flow resistance to the passage of gas due to the extremely small passage space between the liner and the wire contained in it. In any event, the mono-coil construction of the liner causes the injected gas to escape easily from the liner and blend with the mainstream gas. The complexity induced by the mechanical modifications and gas injection variables (flow rate and injector orientation) made the prospect even more cumbersome. Such labor-intensive designs are not readily implemented in field situations. Considering all of these factors, the gas-injection system was reconsidered for design.

It was decided that "automatic injection" at the contact tip end could be achieved by simply terminating the liner approximately 3" above the diffuser and using the gas injector near the tip end as the point for introducing the major portion of the shielding gas. (The main shield gas supply configuration from the wire feeder was not altered). In addition, widening the orifice of the diffuser automatically allowed more gas to flow into the contact tip end. Also, the

proximity of the shielding gas injector to the contact tip enhanced the positive gas pressures prevalent at the contact tip.

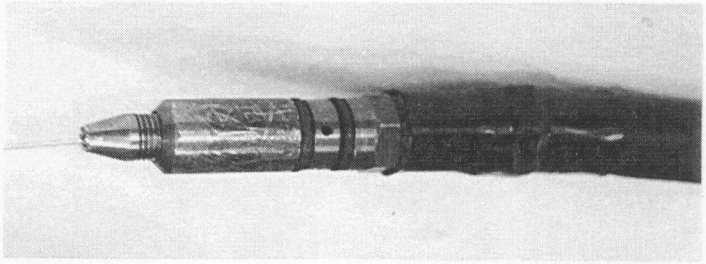
It is important to recognize that the critical point for the ingress of air is not at the contact tip end but at the cable inlet end of a welding torch. At this point, a separate injector was introduced. This injector, located at the wire inlet end (figure 71) efficiently provided flow rates higher than 7 cfh (as opposed to only 0.8 cfh max with a single injector near the contact tip end of the wire liner in the previous design), thus fully ensuring against air ingress.

In order to test the efficiency of the different injection systems, test welds were made on 304 stainless steel plate for each of the following gas shielding configurations:

1. Regular configuration gas shielding without gas injection.
2. Gas injection modification as per described patent (injection into the liner)
3. Dual injection: one injector to pressurize the wire inlet adapter and one injector to pressurize the contact tip area of the torch.

The arc characteristics when welding were not perceived to be different in any way in terms of spatter and sound level. The deposited weld bead appearances did not differ, as testified by figure 72. If any minute variations are indeed produced, they may have to be quantified using appropriate instrumental techniques, such as oscilloscopes and high speed cameras.





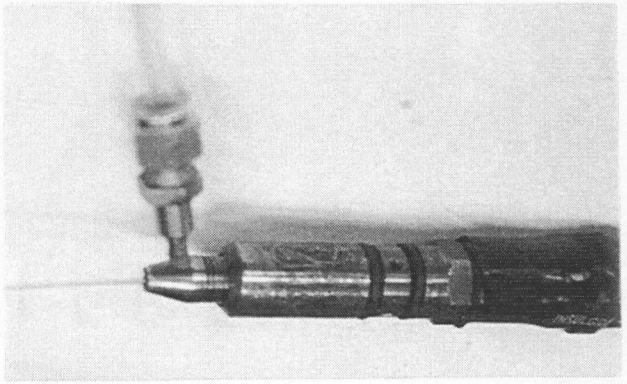


Figure 71. Concept of pressurizing the wire inlet end of the GMA torch cable to prevent air entrainment into the weld pool

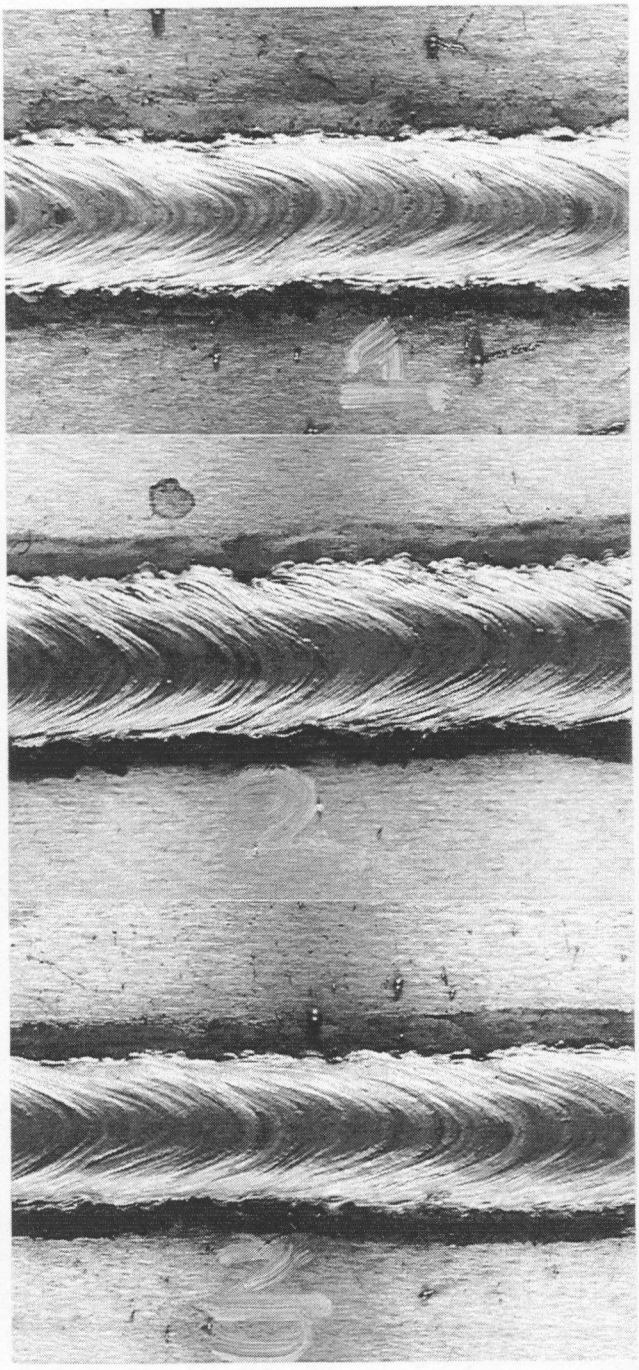


Figure 72. Effect of different shield gas configurations on GMA welded 304 stainless steel. (a) regular shield gas configuration (b) with gas-injection modification as per original patent design (c) with dual-injection

6.15. **Gas Injection: Solid Liner Guns**

The results described in the earlier section proved that trying to achieve gas injection by injecting gas into the monocoil liner/sleeve of a GMA torch (as per patent 5,081,334) is not a viable proposition and is in fact redundant.

Gas injection is theoretically more relevant in GMA torches that utilize a straight, solid liner instead of a mono-coil liner. This is because solid liners, unlike monocoil liners, do not permit leakage of gas. Thus, an injected gas can be expected to partly exit the liner at its two ends, the contact tip end and the wire inlet end. However, the flow resistance is still very high and only about 0.1 % of the injected gas can be expected to exit at the two ends. Tests by Hercules on an injection modified XR gun have confirmed this fact. The Miller XR aluminum welding gun used in the program is an example of a gun that utilizes a solid liner.

Pressure measurement at the tip end of this XR gun indicated that as in the Miller GA-50A torch, a positive gas pressure exists, thus reducing any chances of air entrainment. Apart from the work performed by Hercules using the XR gun, additional work was discovered in the literature that added new light to the concept of gas injection. As far back in the 60's, pressurizing the contact tube and the liner as a means to prevent contamination by ingressed air was an area already researched [69]. In fact, researchers have already used sophisticated infra-red techniques to accurately quantify the extent of impurities with and without pressurization of the guide tube. Pressurization of the contact tube was recommended only as a remedy when excessive contamination seemed to originate from air ingress [70]. Moreover, welds made in the regular

configuration with the XR gun were 100% acceptable and no problems were encountered that could be attributed to possible air entrainment. In fact, injection modification would only complicate the gun configuration and generate risk of wire feed interruption by mechanical fouling of the injector with the moving wire. Besides, duplication of work already performed in the 1960's (work which was never popularized for a probable lack of significance), was a strong motivator in not attempting gas injection in the XR gun. Moreover, all welds obtained with the unmodified gun were fully acceptable and no defects of any sort were detected.

6.16. **Gas Injection: GTA Torch**

Results of the 304 bead on plate GTA welds immediately and conclusively proved that gas injection may not be suitable for GTA torches of this type. The torch configuration is such that the filler wire always enters the arc and weld pool in a direction that cuts the vertical stream of shielding gas. Thus, the interruption caused by an injected gas stream flowing coaxially with the filler wire can only disrupt the regular shield stream, causing shield gas turbulence and subsequent weld degradation. Figure 73 illustrates this fact clearly and brings out the sharp contrast between a weld made in the regular torch shield configuration and a weld made with additional gas injection.

Even at an injected flow rate of 2 cfh, the injected gas apparently caused severe turbulence in the regular shield gas stream. Shielding efficiency dropped to the extent that the weld surfaces produced were clearly degraded.



Figure 73. Deleterious effect of gas-injection in the Astro-arc, automatic GTA torch, on the weld appearance of 304 stainless steel. Weld A-shielded with regular gas shield configuration. Weld B-shielded with gas injection

The weld made with gas injection had an oxidized surface in contrast to the shiny surface of the weld made without gas injection.

Further experiments to alter the injected gas flow rates did not alleviate the deleterious effects caused by injection. It is not likely that any improvement over the regular GTA welds can be obtained using gas injection for torches bearing a similar construction.

CHAPTER 7

CONCLUSIONS

7.1. 304 Stainless Steel: Autogenous GTA Weld Tests

1. The presence of moisture and oxygen in amounts of 320 ppm and 130 ppm, respectively, severely degrades the surface of autogenous GTA welds on 304 stainless steel.

2. The presence of 130 ppm oxygen alone (without any moisture impurity) in the shield gas also visibly oxidizes the fusion zone.

3. Electrode degradation is another consequence of welding with impure shielding gas.

4. Nanochem purification prevents contamination of the weld zone even if the shield gas is grossly contaminated, and safeguards against any deleterious surface effects. The welds shielded with Nanochem-purified gas are clean and shiny, even if the gas is originally grossly contaminated.

5. The bottled gas used in the test was of sufficiently high purity and the weld made with it is comparable to the weld made with Nanochem-purified gas.

7.2. **304 Stainless Steel GMA Weld Tests**

1. Welding without a 2% oxygen additive in the argon shielding gas causes reduced wettability and poor arc characteristics.

2. Considering the necessity of using an active oxygen component in the inert argon shield, the deployment of the Nanochem unit in the system is not suitable when GMA welding 304 stainless steel.

7.3. **316 Stainless Steel: Autogenous GTA Weld Tests**

1. Welding 316 stainless steel with argon contaminated with moisture and oxygen in amounts up to 100 ppm and 300 ppm, respectively, results in visible surface degradation caused by heavy oxidation.

2. Arc wander and a wavy bead are other deleterious effects caused by shield gas impurities in the GTA welding of 316 stainless steel.

3. Nanochem purification of contaminated argon shield gas alleviates the degrading effects seen when welding with contaminated gas. The welds produced are shiny and clean, especially at the weld stop-the area where the evidence of contamination is usually the most easily visible.

4. Nanochem purification of contaminated gas also ensures that the arc does not wander and the resulting bead is straight.

5. Corrosion characteristics in 6% $FeCl_3$ medium are, in general, similar for welds shielded with and without gas purification. The overall corrosion current density is, however, lower for samples welded with purified gas.

6. Welds shielded with bottled gas were comparable to the welds shielded with Nanchem-purified gas, indicating a high shielding gas purity.

7.4. **Titanium: Autogenous GTA Weld Tests.**

1. Titanium welds shielded with the contaminated gas exhibit surface degradation indicated by the formation of blue-colored oxide films along the entire length of the fusion zone.

3. Titanium welds shielded with Nanochem-purified gas exhibit a shiny weld stop and negligible oxide coloration. Nanochem may thus insure against the need for post-weld clean-up operations to remove weld colorations in titanium welds.

4. The weld made with bottled helium also exhibited an appearance comparable to the weld shielded with Nanochem-purified gas, indicating that the purity of the bottled gas was sufficiently high.

5. The bend ductility of autogenous GTA titanium welds is not affected by the shield gas contamination.

7.5. **Aluminum 1100: Autogenous GTA Weld Tests**

1. The presence of moisture in helium-contaminated shields when autogenously welding GTA aluminum causes wavy beads and dull, lusterless weld surfaces with oxide cracks and heavy sooting in the gas coverage region.

2. Purification of moisture-contaminated gas using Nanochem alleviates the situation described above and significantly enhances weld appearance. The welds are shiny and oxide-free.

3. In sea water type test medium, both welds fabricated with and without purification of contaminated gas corroded. Hence, the corrosion characteristics are strongly dependant on the aggressiveness of the service environment. The improved surfaces obtained with Nanochem purification may not provide any benefit in improving the corrosion resistance in a service medium where the welded alloy will inherently corrode.

7.6. **Aluminum 1100: GMA Weld Tests**

1. Moisture and oxygen impurities in argon shielding gas when GMA welding 1100 aluminum results in an overall unacceptable bead appearance characterized by heavy oxide, minor cracks, and rough bead surfaces. The formation of a sooty black deposit in the enitre gas coverage zone is another undersirable effect.

2. Using Nanochem to purify the contaminated gas before welding significantly improves the weld bead appearance. The gas coverage region, although characterized by some oxidation, remains essentially clean and shiny.

3. The shield gas purity may also influence the metal transfer modes slightly. Spray mode was observed with the contaminated gas, while a more globular transfer was observed when welding using purified gas. (Based on computer processed arc sound data).

4. No difference in appearance was found between welds made with bottled argon and welds made with Nanochem-purified, originally contaminated argon. The welding grade gas used was apparently of a high purity. Bottled gas purity consistency, however, cannot always be guaranteed.

5. Bend ductility was not influenced by the purity of the shield gas and was satisfactory and comparable in both types of samples. This behavior is mostly attributed to the inherent ductility of 1100 aluminum and the fact that the effect of impurities was mostly confined to the surface.

7.7. **5086 Aluminum: GMA Weld Tests**

1. The presence of moisture and oxygen or moisture alone in the shielding gas when GMA welding 5086 aluminum alloy with 5556 filler causes severe surface degradation in terms of creating oxide-laden, rough, sooty weld surfaces.

2. The use of Nanochem to purify contaminated gas before welding significantly improves surface appearances. Shield gas purification results in clean, shiny, acceptable welds.

3. The degree of porosity is higher in welds shielded with contaminated gas as opposed to the welds made with purified gas. Purification effectively alleviates the porosity levels in the weld metal.

4. Bend ductility is distinctly improved in welds made with Nanochem-purified helium gas. This improvement may not apply to all shielding gases and gas mixtures.

5. The highly flawed state of the oxide layer produced when welding with contaminated gas jeopardizes the corrosion resistance properties and passivation characteristics in a neutral, chloride-free environment. Nanochem purification of moisture-contaminated helium, on the other hand, significantly improves corrosion resistance properties and passivation characteristics.

6. The benefit derived from purified shielding gas in terms of defect-free, weld oxide films is not able to contribute to improved corrosion resistance in highly acidic, chloride environments.

7.8. **6061 GMA Weld Tests**

1. The presence of moisture in the shielding gas when GMA welding 6061 aluminum alloy with 4043 filler causes visible surface degradation in terms of creating oxide-laden, rough weld surfaces.

2. The use of Nanochem to purify moisture-contaminated helium gas before welding significantly improves surface appearance. Purification results in clean, shiny, acceptable welds.

3. Nanochem purification effectively alleviates the porosity levels by approximately 40% in welds shielded with moisture-contaminated helium.

4. Bend ductility is significantly improved in welds made with Nanochem-purifed helium gas.

5. The highly flawed state of the oxide layer produced when welding with contaminated gas jeopardizes the corrosion resistance properties and passivation characteristics in a neutral, chloride-free environment. Nanochem purification of moisture-contaminated helium, on the other hand, significantly improves corrosion resistance properties and passivation characteristics of the weld.

6. No conclusive explanation is offered to explain why the welds shielded with bottled helium (containing only 16 ppm oxygen and 1 ppm moisture impurity) exhibited poor weld ductility relative to the Nanochem-purified welds.

7.9. **100-S Filler Wire: GMA Weld Tests**

1. The presence of an active gas component in the shield gas when welding with the 100-S GMA solid wire is essential for good arc characteristics and bead appearance. This active shield is either 2% oxygen, as used in the tests conducted, or 5-6% CO_2 as recommended by the wire manufacturer and the U.S. Navy, respectively.

2. Welding 100-S filler wire with inert gas (100% argon) results in poor weld characteristics such as unacceptable degree of spatter and poor weld bead appearance.

3. The use of Nanochem may not be appropriate for welding using the 100-S wire because of its chemical incompatibility with shield gases that contain the recommended active component, namely, 2% oxygen.

7.10. **100-S Filler Wire: GTA Weld Tests**

1. 100-S filler wire GTA weld deposits shielded with oxygen-and moisture-contaminated helium shielding gas have an average diffusible hydrogen content of 5.2 ml/100g.

2. Welding 100-S filler wire using the GTA process shielded with Nanochem-purified gas originally containing approximately 100 ppm oxygen and 300 ppm moisture lowers the average diffusible hydrogen content by approximately 2 ml/100g.

3. In the event that the gas lines in the GTA welding system are saturated with moisture, the diffusible hydrogen level in the 100-S wire weld deposit can be as high as 26 ml/100g.

4. Even if the gas lines are saturated with moisture, the use of Nanochem purification reduces the otherwise inevitable high diffusible hydrogen level to a mere 6.6 ml/100g, a reduction of almost 20 ml/100 g.

5. Arc initiation is difficult when using contaminated helium shields due to increased ionization potential and prior oxidation of the electrode.

6. No arc initiation problems are experienced when using Nanochem-purified shielding gas.

7.11. **AL-6XN Alloy GTA Weld Tests**

7.11.1. **Surface Appearance (Fusion Zone)**

1. Ppm amounts of oxygen and moisture impurities present in the shielding gas cause extensive surface deterioration of autogenous GTA welds of AL-6XN alloy. Weld surfaces produced are dull, rough, and severly oxidized.

2. Welds shielded with Nanochem-purified gas are significantly improved even when the gas is originally grossly contaminated. Weld surfaces are clean, shiny, and show little oxidation.

3. The impurity levels used in the tests were substantial and represent a case for a gross leak in the gas system in an actual welding situation. In the event of such an occurrence, the benefit provided by Nanochem purification has been conclusively proven as a safeguard against weld pool contamination caused by impure shielding gas.

4. Welds shielded with welding grade gas were comparable to the welds shielded with Nanochem-purified gas, indicating a high purity level of the bottled gas. However, inconsistent gas purity and the possibility of rusted cylinders bearing moisture cannot be ruled out in field situations. In such an event, the benefit derivable from using Nanochem purification has been proven and utilization of the Nanochem system is highly recommended.

7.11.2. **Heat Tinted Area**

1. Welds shielded with contaminated gas are extensively heat tinted in the gas coverage zone adjacent to the fusion zone.

2. Nanochem gas purification of contaminated gas results in total elimination of the heat tint.

3. Nanochem purification is recommended for elimination of heat tints caused by impure shielding gas.

7.11.3. **Metallographic Examination**

1. Sub-surface microstructural characteristics (solidification substructure and hardness) are not affected by the level of shield gas purity).

2. The surface tension of the welds are apparently influenced by the impurity level in the gas. The fusion zone area of the spot welds made with impure gas was more irregular, indicating more, random molten pool flow compared to welds made with Nanochem-purified gas.

7.11.4. **SEM Studies**

1. Oxides rich in cerium and lanthanum, found on both types of samples welded with and without purification of contaminated gas, arise from the

deoxidizing alloy additions made during steel-making or from electrode tip wear.

2. The weld surface oxide content of samples welded with contaminated gas is significantly greater and more fragmented compared to welds made with purified gas.

7.11.5. **Corrosion Behavior**

1. AL-6XN welds inherently pit in a 6% ferric chloride medium (as-welded and ground surface conditions) and the corrosion characteristics are not affected by the level of shielding gas purity used in welding.

7.11.5. **Electrochemical Tests**

1. Electrochemical testing in 6% ferric chloride media indicate that welds shielded with Nanochem-purified gas are slightly superior in corrosion resistance characteristics, in both the as-welded and ground surface conditions. The welds are associated with more noble free corroding potentials and lower passive current densities than samples welded with contaminated gas.

2. In 10% sulphuric acid medium, no significant differences are found in samples welded with and without purification. However, heat tints caused by oxidation when welding with contaminated gas lower corrosion resistance characteristics.

3. In 50% boiling sodium hydroxide medium, the corrosion resistance characteristics of welds shielded with Nanochem-purified gas are superior to those of welds shielded with contaminated gas.

7.12. DSS Alloy 255: Filler Added GTA Weld Tests:

1. The oxygen and moisture impurities added to the argon shield gas are mostly swept along with the molten pool under the moving torch. No oxide agglomeration occurs in most part of the fusion zone.

2. However, the agglomerated oxide impurities flowing along with the molten weld pool under the torch solidify immediately after extinction of the arc in the weld crater area. This results in easily observable degradation of the weld crater area.

3. Using Nanochem to purify moisture-and oxygen-contaminated gas when GTA welding without added filler results in a shiny weld and weld crater area.

7.13. DSS 2205 Alloy: GMA Weld Tests

1. Argon-2% oxygen shield gas is advantageous for welding the duplex stainless steels in terms of providing better control of operational characteristics.

2. Fully argon gas shields restrict the control over metal transfer modes and arc characteristics. Weld pool wettability is also reduced.

3. In 6% ferric chloride solution, the overall anodic polarization characteristics of welds (ground condition) shielded with argon-2% oxygen gas, are better than those of welds shielded with argon gas of varied purity levels. There is a tendency, however, for formation of pits at lower potentials.

4. Bend ductility is acceptable and equal in weldments shielded with argon and argon-2%oxygen gases.

5. Nanochem purification of moisture-contaminated argon gas significantly improves toughness properties in duplex 2205 weldments.

6. The presence of up to 300 ppm moisture in argon shield gas does not seem to have any significant deleterious effects on the arc characteristics, bend ductility, corrosion behavior, and toughness properties of duplex 2205 weldments.

7. The use of industry recommended argon-2% oxygen gas significantly degrades the toughness properties of duplex 2205 weldments.

7.14. **Gas Injection: GMA Torch**

1. There does not exist any need for trying to pressurize the wire liner (gas-injection) in the GA-50 A torch to prevent air ingress. The monocoil nature of the torch with its continuous "holes" and the positive pressures at the wire inlet and tip end already attest to the positive pressures prevalent in the liner. This situation automatically prevents air ingress.

2. The high flow resistance due to the long length of the liner and its monocoil construction render impossible any attempts to inject gas into it.

3. "Dual-Injection", i.e. pressurization of the wire inlet adapter in addition to pressurization of the contact tip area of a GMA torch cable , is a more effective means to reinforce the positive pressures already prevalent at the wire inlet end, thus safely ensuring against air entrainment.

4. Automatic and effective pressurization of the contact tip end can be achieved by terminating the liner an inch away from the gas diffuser end.

5. The concept of guide tube pressurization to prevent air ingress in GMA torches is not new and was conceived as early as 1960.

7.15. **Gas Injection: GTA Torch**

1. The gas injection modification on a torch of this nature disrupts the regular shield and results in degraded weld surfaces.

2. Gas injection modifications in torches where the wire feed tip is oriented so as to cut across the regular shield stream are not recommended.

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