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Collider Accelerator Department

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RHIC PROJECT

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WELDING CONSUMABLE SELECTION FOR A CRYOGENIC (4 K) APPLICATION

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ABSTRACT

This paper provides a brief description of the development and qualification of an appropriate welding consumable for a demanding cryogenic magnet application. It began with a search through the research conducted in the past decade on cryogenic fracture toughness of wrought and welded austenitic stainless steels. This research shows that certain elements of the composition have a powerful effect upon the steel's fracture toughness at 4 K. In particular, the higher oxygen in the weld manifests itself as inclusions, which have a severe detrimental effect upon the fracture toughness. This one factor accounts for most of the difference in toughness between matching composition wrought and weld material, and is a function of the weld process. Also, welds enriched with manganese and nickel have demonstrated improved fracture toughness. These discoveries were combined in the development of a nitrogen and manganese modified, high nickel stainless steel alloy. It produced gas metal arc welds with superior cryogenic mechanical properties (yield strength near 900 MPa at 4 K and a Charpy V-Notch impact energy near 140 J at 76 K), when the procedures were modified to reduce the oxygen content.

BACKGROUND

In 1983 and in 1989, the nuclear physics scientific community prepared long-range plans for the U.S. Department of Energy and the National Science Foundation. In both years, they identified the Relativistic Heavy Ion Collider (RHIC) as the highest priority for facility construction. In response, the U.S. Congress appropriated the first construction funds for RHIC in Fiscal Year 1991. The construction of RHIC, at the Brookhaven National Laboratory (BNL), will provide the United States with a world-class facility with a potential for unique discoveries. Specifically, RHIC will be able to create matter at extremely high temperatures and densities — so extreme that scientists hope to observe phenomena that have not occurred in the natural universe since the original "Big Bang." These experiments cannot be conducted at existing high energy accelerator facilities.

In RHIC, two beams of heavy ions will speed in opposite directions inside a pair of rings in a tunnel almost 3.9 kilometers in circumference. The beams will be bent and focused by over 1700 superconducting magnets. The material and magnetic property requirements (material strength to resist the mechanical loads and the fracture toughness requirements at 4 K) of these magnets are very demanding. This paper concentrates on the weld design and materials to meet the design requirements of the superconducting magnet structures.

BNL Magnet Design Requirements

A superconducting magnet has the same basic structure as a traditional electromagnet — a wound electrical cable with an iron core. In the case of niobium-tin superconducting magnets, the electrical cable is superconducting and must be maintained at a temperature less than 4.6 K. The cable and iron core are wrapped within a cryogenic pressure vessel to provide this cooling. The U.S. Department of Energy requires that all pressure vessels at its facilities comply with the

American Society of Mechanical Engineers Boiler & Pressure Vessel Code. These requirements, coupled with the cryogenic system design and manufacturing cost parameters, required a 900 MPa weld yield strength for the 4.8-mm-thick stainless steel magnet shells, a FN less than 3 for weld processes other than GTAW and GMAW, and a lateral expansion greater than 0.38 mm for a Charpy V-Notch (CVN) impact test at the operating temperature. ASME Code paragraph UG-84 requires impact testing for applications operating at temperatures below 19 K, where the RHIC pressure vessels will be operating, yet impact testing at 4 K is complicated by the low heat capacity of materials at this temperature. The ASME Code (originally developed for applications near room temperature or above) had been applied to cryogenic temperatures without adjusting for the changes in the physics of heat flow and deformation. The standard Charpy impact test prescribed by ASTM Standard E23 relies upon the room temperature properties of materials. A study by Tobler shows that a specimen cannot be transferred from a 4 K cooling bath to the test machine quick enough to avoid exceeding the test temperature by a large margin.[Ref. 1] Furthermore, adiabatic heating has such an influence on all materials at this temperature that the deformation preceding fracture often produces a 70 K increase in specimen temperature. This means that the specimen temperature might far exceed the test temperature during fracture even while using innovative techniques to keep the specimen cool until the instant of fracture (such as cooling it while already sitting on the impact machine anvils). This testing may provide acceptable data for projectile impact tests, but cannot provide valid data for a large structure at 4 K. What we wanted was a large cryogenic structure that would not fracture, not simply good impact data. The solution to this dilemma is an engineered weld.

Engineered Weld

Fracture mechanics calculations conducted for several cases showed that the fracture toughness (K_{Ic}) of the magnet should be at least 68 MPa√m. While this fracture toughness requirement appears relatively easy to achieve, it forms the *minimum* design requirement. Current data indicates a one sigma scatter band of +/- 44 MPa√m.[Ref. 2] This indicates the nominal fracture toughness value must be at least 156 MPa√m to guarantee a 95% confidence level is achieved. This requirement places the necessary fracture toughness at the upper boundary for weld metal, as shown in Figure 1.

One complexity in designing for fracture is that many published reports and the ASME Code specify CVN impact requirements as absorbed energy, in Joules, or lateral expansion, in mm, while strict fracture mechanics calculations use fracture toughness data, in MPa√m. These are significantly different approaches and we wanted to be certain that we fulfilled both needs. Therefore, we generated both types of data for our welds, then compared our data to both types of requirements.

Conventional wisdom says the weld should match the composition of the base material. This general rule very often is useful in helping designers to avoid problems due to thermal expansion differences (residual stress and distortion), corrosion potentials, and strength differences. This structure is not subject to a severe corrosion environment, but the other problems could be important, so we favored a matching composition. To guide our selection of the electrode composition, we found several studies of materials and joining processes for specific cryogenic magnet structures that provided very practical advice. [Refs. 3 and 4] In particular, the 1985 paper by Goodwin describes the construction of large, type 316LN stainless steel magnet cases (the Large Coil Program) for 4 K service. He found an extremely wide range in reported mechanical properties for candidate welding consumables and describes how they qualified electrodes that met their property requirements. For RHIC, we wanted a better margin between the requirements and typical properties than those listed in this study (for greater reliability), and so we considered alternate compositions, especially ones developed or evaluated since these reports.

Other published studies of material properties provide broader guidance on the effect of various elements and on the selection of a material to meet a set of mechanical properties.[Refs. 5 to 18] Reference 14 shows the strength of austenitic stainless steel at cryogenic temperature is controlled primarily by the nitrogen content. Predictive equations for weld strength have a relatively small scatter (standard deviation near 50 MPa, or about 6% for this application) and the range of strength data spans the 900 MPa goal of RHIC. Figure 1 shows strength versus toughness data for types 308L and 316L stainless steel compositions. Unfortunately, it shows that as the strength increases (through nitrogen additions), the toughness decreases, so we were concerned about our ability to meet the strength and the toughness requirements simultaneously. Figure 1 also shows that the welds fall short of the base metal properties. This was of concern to the designers because welds tend to have an uneven surface profile and usually contain high residual stresses which can grow still higher when cooled to cryogenic temperatures. The combination of lower toughness, surface roughness, and residual stresses could make the welds a critical fracture path for an unexpected tensile overload or fatigue cracks. We were looking for welds that could match both the strength and toughness of the 304L base material.

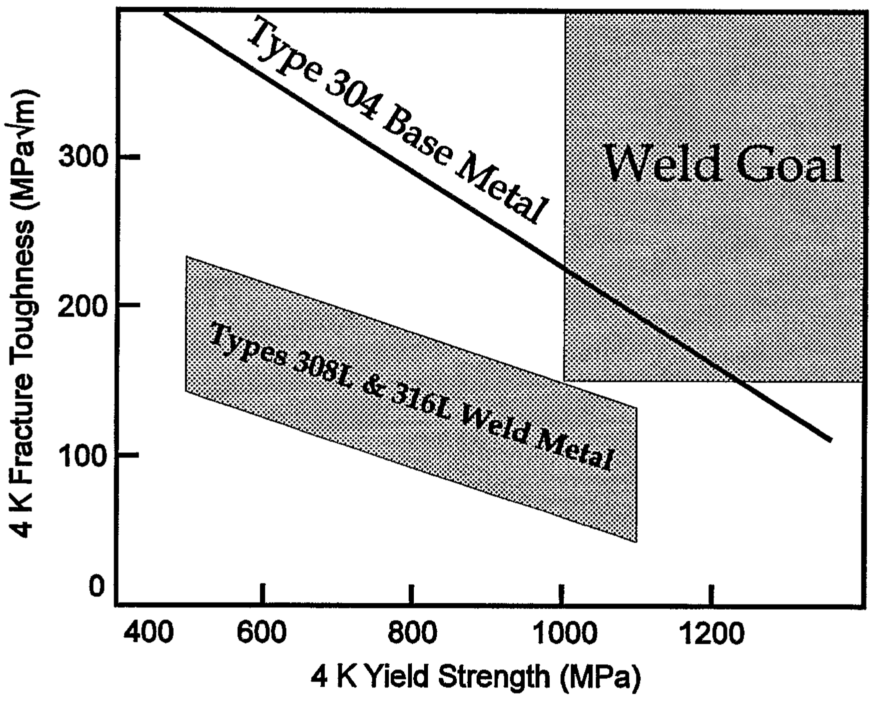

Figure 1
Comparison of the Weld and Base Metal Strength-Toughness Relationship

FACTORS AFFECTING CRYOGENIC STRENGTH & TOUGHNESS IN WELD METAL

Weld toughness is affected by many factors. Metallurgical factors causing low toughness in weld metal are well known to be precipitates such as carbides, nitrides, and intermetallic compounds. The presence of delta ferrite and non-metallic inclusions also are well known detractors to toughness. Typical compositions (types 308 and 316) used for cryogenic stainless welds generally fall about 40% below base metals in their σ_y versus K_{Ic} performance (See also Figure 1).[Ref. 2]

Ferrite

Delta ferrite is a residual phase present in some stainless steel welds that solidify in a primary ferrite mode. Residual ferrite in small quantities is normally desirable in stainless steel welds because it inhibits the formation of low melting point compounds (such as FeS and FeP) which promote hot cracking in fully austenitic alloys. However, ferrite should be minimized for best toughness in cryogenic service. Therefore, welding alloys for cryogenic service are either ferrite-free or very low ferrite. The ferrite-free alloys are produced with very strict controls on the impurity contents that promote hot cracking.

NIST data show an inverse relationship between yield strength and fracture toughness.[Ref. 5] Also, welds with a Ferrite Number (FN) greater than 7 show relatively low toughness, but welds with a lower FN are scattered within a one sigma scatter band of +/- 44 MPa. [Ref. 2] This is shown graphically in Figure 1. Thus ferrite should be reduced to the lowest level consistent with fissure resistance. Test data available to date indicates the strength-toughness characteristics of welds may be increased by eliminating delta ferrite, avoiding chromium carbides, and reducing the width of columnar grains. These actions will raise the trend line of the weld strength-toughness characteristics closer to that of wrought stainless steel. Other research establishes a Charpy absorbed energy of at least 32 Joules is necessary to meet the ASME minimum lateral expansion of 0.015 inches (0.38 mm).[Ref. 6] Reference 7 lists an equation for 76 K CVN impact energy as a function of FN, calculated from the Schaeffler Diagram (ferrite potential if negative), carbon content, and nickel content.

$$CVN\,(J) = 19 - 1.4FN - 890C^2 + 1.4Ni$$

This equation indicates a CVN of 32 Joules is not possible with FN greater than 2, carbon content greater than 0.03 wt.%, and nickel content less than 12 wt.%.

Nitrogen

The strengthening characteristics of nitrogen becomes more pronounced at lower temperatures. Figure 2 shows how 316L weld strength increases by a factor of 2 as temperature is decreased from 298 K to 76 K, and increases by a factor of 2.5 at 4 K for the same 0.05% nitrogen. But increasing the nitrogen from 0.05% to 0.20% yields a three-fold increase in strength when cooled to 4 K.[Ref. 7]

Nitrogen cannot be added without limit. The upper limit is determined by the solubility of nitrogen in the microstructure, above which weld porosity results. The solubility limit is a function of the composition, with certain elements (such as manganese) serving to increase the limit. [Ref. 8] Although porosity is not as deleterious to the mechanical properties as a crack, we decided to add manganese above the 1.5 wt.% typical for austenitic stainless steels, to provide greater protection from porosity formation, as well as act as a solid solution strengthener.

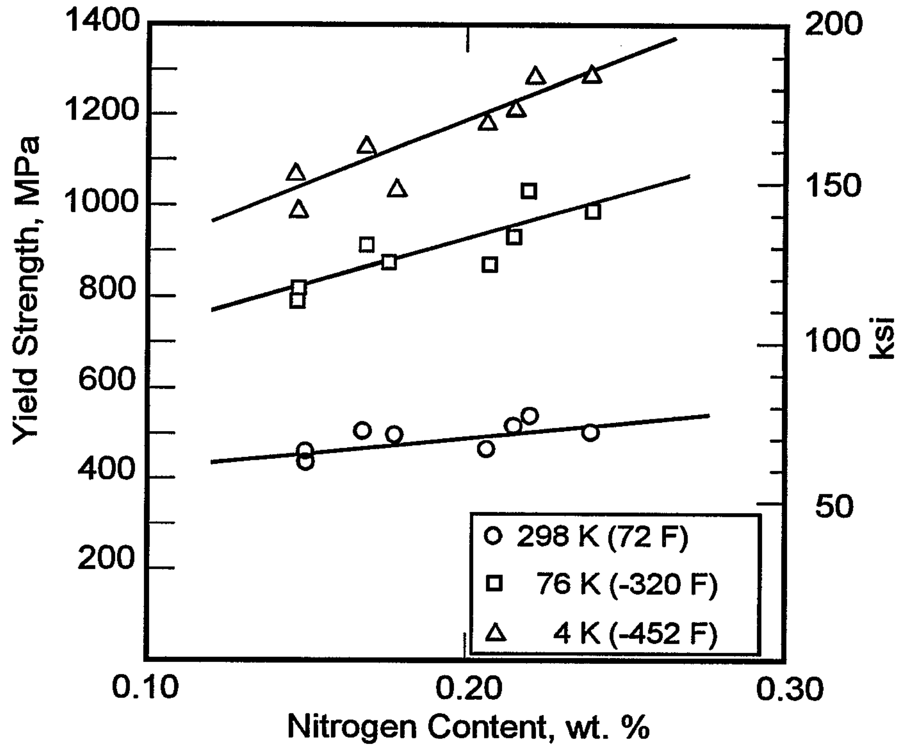

Figure 2
Yield Strength versus Nitrogen Content for Type 316LN Welds

Nickel

Nickel also has a significant, though nonlinear, effect on toughness. Figure 3 shows that increasing nickel from 10% to 20% provides the greatest improvement in toughness. Thus, a weld with 20% nickel should exhibit the greatest attainable toughness for an austenitic stainless steel. NIST had performed work to determine the best weld material for 316LN, which is a popular base material for demanding cryogenic applications.[Ref. 7] Two commercially available compositions - 18Cr-20Ni-5Mn-0.16N and 20Cr-25Ni-4.5Mo — were evaluated using gas metal arc welding. Shielding gases were more inert than normal to reduce oxygen content, and the gas used for the 20Cr-25Ni-4.5Mo electrode was augmented with nitrogen to increase the nitrogen content of the weld metal. Figure 4 shows that the strength was comparable to 316LN base metal and the fracture toughness was as high as or exceeded that of the 316LN base metal. This toughness is clearly higher than the toughness achievable with 308 and 316-based welding compositions and standard welding procedures.

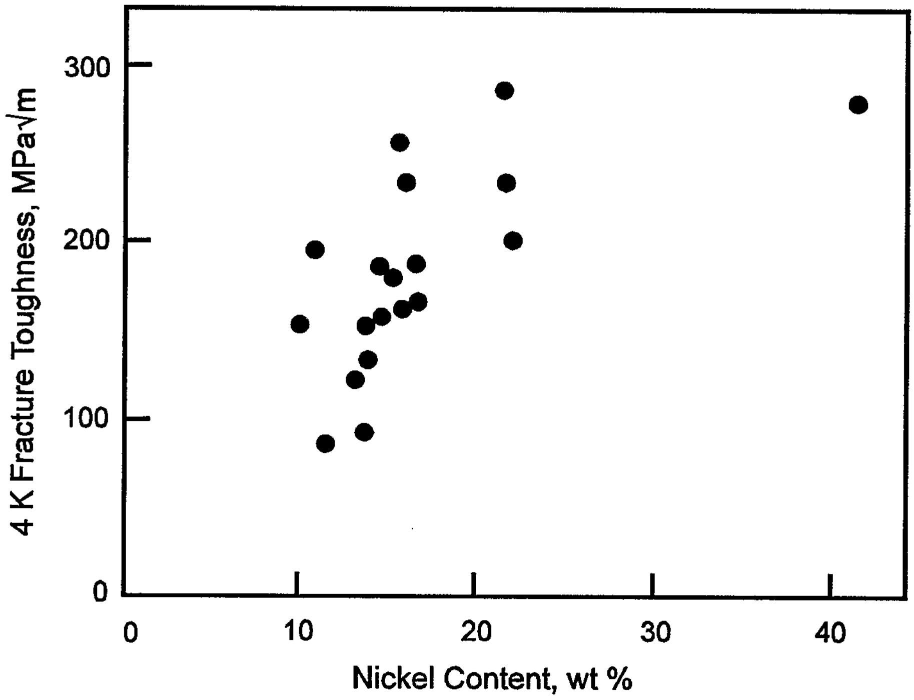

Figure 3
Fracture Toughness versus Nickel Content

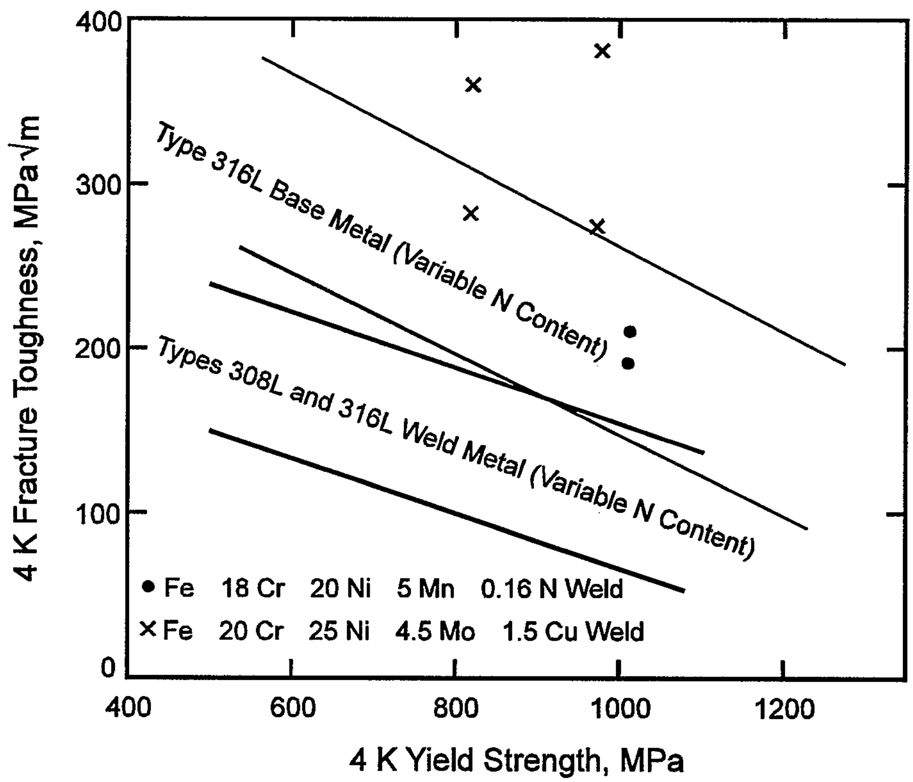

Figure 4
Fracture Toughness versus Yield Strength

Other research shows that higher nickel contents improve the toughness in two ways: nickel reduces the weld metal ferrite content (a magnetic microstructural phase and more brittle than austenite), and nickel additions increase the toughness in fully austenitic compositions.[Refs. 9, 10, 11 and 12] Figure 3 shows how nickel additions to stainless steel alloys generally increase the toughness, at least up to 20 wt.%. A secondary benefit of nickel is that it stabilizes austenitic structure against the formation of martensite (another magnetic phase) during deformation of the structure. The two references on the fabrication of the cryogenic magnet structures mentioned their search for these magnetic phases during welding procedure qualification and control.[Refs. 3 and 4]

Tramp Elements

Fully austenitic compositions may be subject to hot cracks, which are ruptures that form in the hot weld during, or just after, solidification. Studies show that this tendency can be controlled by careful control of elements that produce a low-melting-point eutectic.[Ref. 13] This problem is evident in electrode specifications where crack-sensitive compositions can be available in a special grade with stricter limits on the elements that promote hot cracking. Sometimes the fully austenitic grades can be made less crack sensitive by adding elements (such as manganese, copper, or carbon) that change the solidification structure. These elements may change the shape of the solidification front or change the amount of eutectic between adjacent dendrites.

Oxygen

Because of its reactivity, oxygen is not found in the free state in the weld, but combines with other elements to form oxide inclusions. These inclusions have diameters near one micrometer and are spherical in shape because they form in the liquid above the solidification temperature. As the weld cools, these inclusions are entrained in the solid and have little effect on mechanical properties until the weld is deformed. Inclusions are harder than the surrounding metal matrix, serving as impediments to the plastic flow of atoms during deformation. As a result, substantial stresses form in the vicinity of inclusions causing voids to initiate. These voids then link by void coalescence, leading to final fracture of the material. Since the voids nucleate at inclusions, reduction in the inclusion density and size reduces the number of voids that form, and is an obvious step in increasing the toughness of a weld. Stainless steel welds tend to have a lower toughness than wrought material. The sources of the difference between weld material and wrought material are the inclusion and ferrite contents. Welds will have a higher inclusion content because of the imperfect gas shielding of the metal while molten. Studies have shown that toughness correlation with inclusion spacing is similar for wrought material, thus attributing the differences in fracture toughness to this one factor.[Refs. 14 and 15]

Research on the effects of the welding process and shielding gas on toughness has been conducted using 308, 308L, and 316L filler metal, gas tungsten arc welding (GTAW) with 100% argon shielding or gas metal arc welding (GMAW) with Ar/2% O_2 and Ar/5%O_2.[Ref. 6] The results are shown in Figure 5. The Type 316L weld metal chemical composition was consistent using the different processes, but differed drastically in oxygen content (0.005 - 0.076%). Lateral expansion properties would meet ASME requirements at 173 K, but only the GTAW weld shielded with pure argon would meet the requirement at 76 K.

Type 308/308L weld metal oxygen content ranged from 0.007 to 0.15%, and were similarly affected by oxygen content, with steep declines in lateral expansion and impact energy. Impact properties began to stabilize when oxygen content reached 0.06% oxygen. Mechanical tests revealed a relationship between FN and oxygen content, and the ability to meet the ASME lateral expansion requirement. However, it was found that the low oxygen GTAW weld could meet the ASME requirement quite safety even with a relatively high FN, while the high oxygen content welds cannot meet the lateral expansion requirement even with a FN of 5. This accounts for the ASME recommendation for a FN lower than 3 for weldments other than GTAW and GMAW.[Ref. 16]

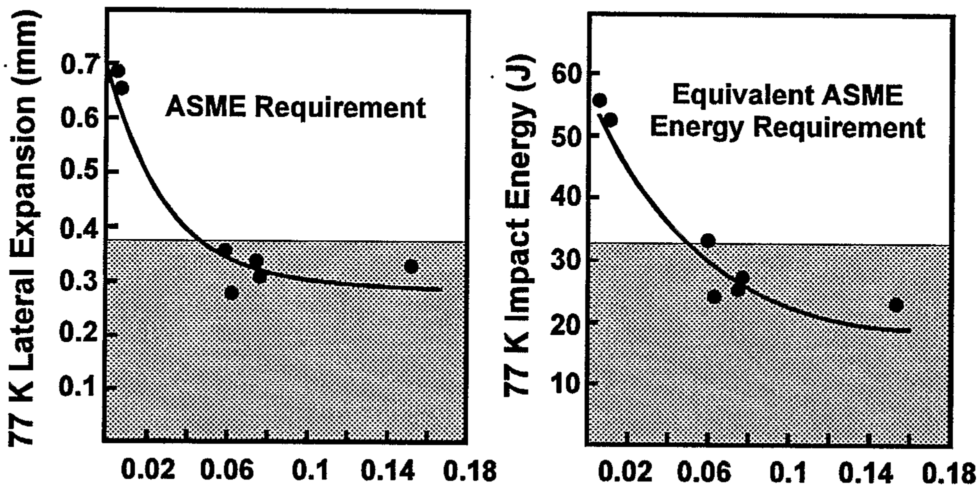

Figure 5
Results of Oxygen Content Investigation[6]

The study by Kim also found an excellent correlation between lateral expansion and impact energy at 173 K and 76 K.[Ref. 6] This relationship is described by:

$$LE(mm) = 0.12 \times C_V(Joule)$$

Thus, the ASME lateral expansion requirement is equivalent to an impact energy of 32 Joules (23.6 foot-pounds). Fractographic analysis of the 76 K Charpy V-Notch specimens showed more brittle fracture of ferrite on the fracture surface of samples with decreased oxygen content. This is attributed to initiation and propagation for welds having retained delta ferrite and very low inclusion content. The fracture in this case appears to be more likely to initiate in and follow the ferrite phase. The overall fracture process, however, still requires high energy, because the ductile austenitic matrix prevents continuous brittle fracture in the weld. High oxygen weld fractures initiate by the formation of microvoids and propagate by microvoid coalescence. This proceeds so easily that the whole fracture occurs in a fully dimpled mode at low energy. Therefore, it is possible to increase impact toughness in low oxygen welds by decreasing ferrite, but ferrite control would not be effective in high oxygen welds because ferrite has a negligible role in the fracture process.

This effect also was studied by Whipple and Kotecki, who produced a series of 316L welds using GTAW, GMAW, and submerged arc welding (SAW).[Ref. 17] The toughness at 4 K was found to be inversely proportional to the inclusion content, with the highest toughness found in the GTAW welds (181 MPa√m). Other research found the 4 K K_{IC} of Type 316L stainless steel weld composition increased significantly when inclusion contents in GMAW welds were decreased.[Ref. 18] The study showed an increase in toughness of 18 MPa√m per micron increase in average inclusion spacing. Siewert and McCowan's study used specimens made by varying shielding gas composition over 304 plate with 316L electrode.[Ref. 18] Material properties are shown in Table 1. Inclusion density had little effect on yield strength, which varied less than 4%, but fracture toughness increased by 35% as the inclusion content decreased by 65%. The wide scatter for toughness data of weld metals is attributed to the varying inclusion contents when several welding processes are used.

Finally, it has been shown that the inclusion density is linearly related to oxygen content, as shown in Figure 6.[Refs. 6 and 15] This is good news because oxygen content can be determined much more quickly and economically, using standard procedures and equipment, than inclusion density, for which standards have not yet been developed. Clearly, minimizing oxygen content, thus minimizing inclusion density, will assure that a minimum toughness will be exceeded.

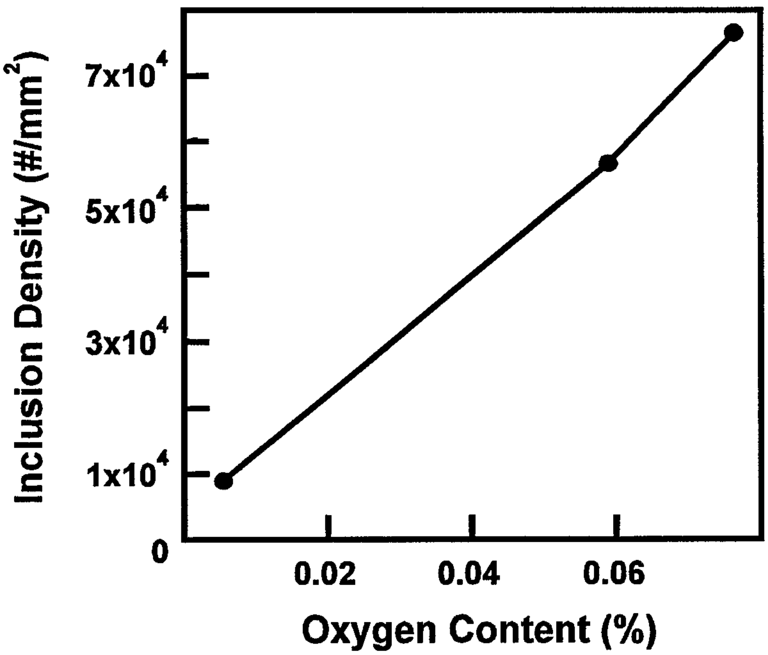

Figure 6
Inclusion Effect on Impact Toughness[6]

Table 1
Inclusion Effect on Inclusion Density and Yield Strength

σ_y (MPa)	El (%)	RA (%)	K_{Ic} (MPa$\sqrt{m}$)	Oxygen (wt %)	Inclusions (#x10^3/mm^2)	Inclusion Spacing (μm)
736	47.9	46.6	179	0.004	19,300	7.0
747	22.3	23.7	150	0.048	37,700	5.0
743	10.2	13.1	132	0.072	55,200	4.3

Final Electrode Composition

By combining the desirable ranges for the various elements listed above, we arrive at the following composition for our weld (and welding electrode):

- 25 wt.% nickel (to provide good toughness),
- 20 wt.% chromium (to develop the fully austenitic composition),
- 0.018 wt.% nitrogen (to provide the 900 MPa yield strength at 4 K),
- 7 wt.% manganese (to increase the solubility of nitrogen, strengthen the weld, and reduce the hot cracking sensitivity),
- 1.5 wt.% copper (to reduce the hot cracking sensitivity),
- 5 wt.% molybdenum (to strengthen the weld),
- 0.005 wt.% upper limit on phosphorus and sulfur (to reduce the hot cracking sensitivity), and
- 0.02 wt.% upper limit on oxygen (to produce higher toughness).

Working with electrode manufacturers, we adopted the composition specification detailed in Table 2.

Table 2
Electrode Specification [19]

Element	Range (%)
Carbon	0.02 max
Manganese	7.0 - 7.2
Silicon	0.2 - 0.5
Phosphorous	0.018 max (desired as low as possible)
Sulfur	0.004 max (desired as low as possible)
Chromium	20.9 - 21.7
Nickel	24.75 - 25.25
Molybdenum	4.75 - 5.25
Copper	1.25 - 1.75
Nitrogen	0.17 - 0.21
Oxygen	0.015 max (desired as low as possible)
Other	< 0.50
Iron	Remainder

Because this composition does not match that of the Type 304L base metal, we need to reconsider the potential problems of nonmatching compositions. This composition is fully austenitic, as is the base material, so they should have closely matched coefficients of thermal expansion, and no residual stress problems. The strength has been matched, so there should not be unbalanced strain problems. The weld has a high nickel content, so it should have no ferrite and be very resistant to martensitic formation under deformation.

Process Selection

The differences between weld material and wrought material are the inclusion and ferrite contents. Welds will have a higher inclusion content because of the imperfect shielding of the metal while molten. Therefore choosing the welding process that produces the lowest inclusion content or modifying the process to reduce inclusion content is required to improve weld toughness. Welding processes such as laser, electron beam and gas tungsten arc welding (GTAW) can produce welds with lower inclusion contents and produce welds with toughness at the upper side of the scatter band (Figure 1).[Refs. 17 and 20]

Initially, we considered a variety of welding processes — shielded metal arc, gas tungsten arc, gas metal arc, and flux cored arc — because they all are appropriate for base metals with a thickness near 5 mm, and do not require special chambers, special alignment, or expensive power sources. From this list, we eliminated shielded metal arc welding because it is not amenable to automation, and ranked gas tungsten arc welding lower than the others because it has a lower deposition rate. Keeping several processes on the list gave us some options as we began a search for the best composition.

The number of inclusions is a function of both the oxygen content of the welding electrode as manufactured, and of the oxygen that is added during welding. The oxygen content must be controlled during both of these times in order to produce the best toughness in the weld. We were unable to locate a flux cored arc welding slag system that produced low inclusion contents, so this reduced our weld process choices to gas metal arc and gas tungsten arc welding.

Gas metal arc welding is preferred for higher production rates, but the process might not produce adequate mechanical properties unless tightly controlled. Some of the newer pulse power supplies that are commercially available provide excellent manual welding results. Other, older power sources are less flexible because their preset schedules are not applicable to the wide range of filler materials and gas compositions. BNL selected a modern weld power source employing a proprietary, constant-current power supply with a patented, pulsed-width modulated, constant-voltage control. This feature provides the ability to optimize the pulsed spray GMAW arc and process characteristics using a set of direct unit controls. The full range of parameter controls on this system are far more complicated than the typical single-knob systems, but the process is more suitable for automated operation. More precise control over the arc and other process characteristics yield a cleaner weld with more consistent composition and microstructure.

TEST PROCEDURES

Materials and Welding Details

Two heats of this composition were ordered. NIST ordered a small laboratory heat (100 kg) to evaluate the properties. Upon successful results with this laboratory heat, BNL ordered a production quantity and evaluated it in a similar manner. The chemical compositions used in this study, as received from the manufacturer, are shown in Table 3.

The testing was conducted in three increments. NIST determined the mechanical properties of the alloy and the effect of oxygen on the weld metal. BNL first evaluated the NIST alloy to establish a baseline for material properties, then evaluated its own heat to verify mechanical properties. All welds were deposited using the gas metal arc process in single V-grooves, as specified in AWS A5.5-81, on Type 304 base plates; 25-mm thick for all NIST testing, 12.7-mm

Table 3
Tested Compositions

	C	Mn	Si	P	S	Cr	Ni	Mo	Cu	N	O
NIST	0.013	7.22	0.39	<0.005	0.0019	21.36	25.16	5.16	1.61	0.19	0.0048
BNL	0.013	7.2	0.4	0.015	<0.001	21.3	24.97	4.86	1.25	0.19	0.002

for BNL Charpy testing, and 4.8-mm thick for BNL tensile testing. Test weld parameters are listed in Table 4.

Table 4
Test Weld Parameters

Alloy/ Test	Shielding Gas	Current (A)	Voltage (V)	Travel Speed (mm/s)	Heat Input kJ/mm)	Wire Dia. (mm)
NIST	Ar	280	31	3.5	2.5	1.2
NIST	Ar/0.5% O_2	280	30	3.4	2.5	1.2
NIST	Ar/2% O_2	270	31	3.4	2.5	1.2
NIST/ BNL Charpy	Ar	212	28	2.5	2.4	1.2
NIST/ BNL Tensile	Ar	150	28	3.5	1.2	1.2
BNL/ BNL Charpy	Ar/1% CO_2	175	22.5	6.1	0.64	0.9
BNL/ BNL Tensile	Ar/1% CO_2	175	22.5	6.1	0.64	0.9

One of the purposes of the NIST testing was to evaluate the effects of inclusions. Within each series of NIST welds, systematic variations in the weld metal inclusion volume fraction were achieved by varying the oxygen potential of the shielding gas. For these welds the heat input was kept fairly constant at 2.5 kJ/mm in order to obtain a relatively equal inclusion size distribution. These welds were performed manually, with a shielding gas flow rate of 16.5 l/min. The initial BNL test welds using the NIST alloy were performed manually, using laboratory pure (99.999%) argon flowing at 16.1 l/min. Past experiences with commercial grade argon led BNL to the adoption of laboratory grade for all welding. However, government rates for this grade remain below the non-government cost for commercial grade, resulting in a negligible cost impact. The BNL test welds using the BNL alloy were performed automatically using a pulsed gas metal arc process. Shielding gas was laboratory pure argon with 1% carbon dioxide, flowing at 16.5 l/min. The addition of carbon dioxide was necessary for arc stability.

Mechanical Testing

From each NIST weld, one all-weld-metal uniaxial tensile specimen (6.25-mm diameter, 25.4-mm-gauge length) and two through-thickness compact tension specimens (CTS) were machined for testing in liquid helium (4 K). The CTS were tested in accordance with ASTM Standard E 813-89, using the single-specimen compliance method, while the tensile specimens were strained at a constant cross head speed of 0.5 mm/min. The BNL magnet design employs 4.8-mm-thick shells, hence tensile test specimens were transverse specimens machined from 4.8-mm-thick welded test plates with the weld centered within the gage length. The tensile specimens were strained at a constant cross head speed of 10 mm/min. Charpy impact testing was performed in accordance with ASTM Standard E 23-88 with full-size V-notch specimens using a machine with a U-shaped pendulum and 325 Joule capacity.

The American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code requires testing of the mechanical properties at the service conditions. Tensile testing at 4 K requires special equipment, but the procedures are well documented. We followed the procedure of Tobler et al. of chilling the specimens, adding liquid helium until they are submerged, and performing the test while in equilibrium with the boiling helium.[Ref. 21] Similarly, the Charpy specimens were cooled to 76 K using liquid nitrogen following the procedures for liquid cooling in ASTM Standard E 23-88.

Metallographic Examination

The NIST welds were sectioned transverse to the welding direction and prepared using standard grinding and polishing techniques for metallographic examination. The two-dimensional (2-D) inclusion volume fraction and size distribution were determined by scanning electron microscope in combination with automatic image analysis at a magnification of 2500 X, using polished specimens. A total of 500 particles were counted for each specimen. In these measurements, due care was taken to ensure that particles present immediately beneath the metal surface were discriminated through proper adjustment of the microscope operating parameters.[Ref. 22] The BNL welds were similarly prepared, but image analysis was conducted at 2000 X with an optical microscope.

TEST RESULTS

Inclusion Characteristics

The measured two-dimensional (2-D) inclusion diameters and the number of inclusions per unit area are shown in Table 5 and compared with the oxygen content for the test welds. The Alloy/Weld column reflects the alloy composition (Table 3) and agency performing the weld. The inclusion density has a direct relationship with oxygen content, with a 25% reduction in oxygen content producing a 36% reduction in inclusion density, and a 50% reduction in oxygen content producing a 50% reduction in inclusion density. Although the BNL welds, overall, experienced higher inclusion densities, this is attributed to the difference in procedures and equipment. Most significant in this study is that inclusion density remained directly proportional to the oxygen content. Oxygen content can be obtained more quickly and easily than inclusion density or Charpy tests, with techniques less prone to errors. Thus, relating the desired fracture toughness to oxygen content would be a desirable future goal, but requires validation for a wider range of materials and processes.

Table 5
Test Weld Inclusion Size Distribution and Density

Alloy/Weld	Shielding Gas	O_2 Content (wt %)	Density ($\#x10^3/mm^2$)	Mean Dia. (μm)	Std. Dev. (μm)	Max. (μm)
NIST/NIST	Ar	0.0121	2.21	0.35	—	
NIST/NIST	Ar/0.5% O_2	0.0231	4.80	0.33	—	
NIST/NIST	Ar/2% O_2	0.0463	8.46	0.35	—	
NIST/BNL	Ar	0.024	17.45	0.415	0.232	1.4
BNL/BNL	Ar/1% CO_2	0.018	11.20	0.399	0.208	2.1

A previous study showed the inclusion density is strongly affected by an increase in the heat input. These results are demonstrated here, as well, with a 47% reduction in heat input producing a 36% reduction in inclusion density.[Ref. 23]

Weld Metal Tensile Properties

The results of the 4 K tensile testing are summarized in Table 6. The 0.2% offset yield strength ranged from 868 MPa to 995 MPa, and the ultimate tensile strength ranged from 1222 MPa to 1412 MPa. In addition, a minimum value of 26% has been obtained for elongation at fracture and 24% for reduction of area.

Table 6
Weld Metal 4 K Tensile Properties

Alloy/Weld	Shielding Gas	Sy (MPa)	Su (MPa)	Elongation (%)	Reduction of Area (%)
NIST/NIST	Ar	995	1337	49	42
NIST/NIST	Ar/0.5% O_2	973	1222	26	24
NIST/NIST	Ar/2% O_2	940	1271	39	27
NIST/BNL	Ar	874	1319	41	—
BNL/BNL	Ar/1% CO_2	868	1412	31	27

Weld Metal Toughness

The fracture toughness results ranged from 218 MPa√m to 286 MPa√m, and are listed in Table 7. These results are discussed elsewhere, but notice there is considerable reduction in fracture toughness with increasing inclusion density and oxygen content.[Ref. 23] For this study, Charpy V-Notch testing was conducted at three temperatures, with the results shown in Table 8. CVN energies at 76 K ranged from 136 Joules to 174 Joules, and room temperature energies from 193 Joules to 243 Joules. CVN energies increased an average of 25 Joules with a 25% reduction in oxygen content, or a 36% reduction in inclusion density. The results also are plotted in Figure 7.

Table 7
Weld Metal 4 K Fracture Toughness

Alloy/Weld	Shielding Gas	O_2 (Wt. %)	Incl.Dens. (#x10^3/mm^2)	KQ (MPa√m)
NIST/NIST	Ar	0.0121	2.21	272
NIST/NIST	Ar/0.5% O_2	0.0231	4.80	286
NIST/NIST	Ar/2% O_2	0.0463	8.46	218

Table 8
Weld Metal 4 K Fracture Toughness

		295 K		185 K		76 K	
Alloy	O_2 (%)	CVN (J)	LE (mm)	CVN (J)	LE (mm)	CVN (J)	LE (mm)
NIST	0.024	209	2.27	176	2.07	139	1.52
BNL	0.018	229	1.58	206	1.53	166	1.21

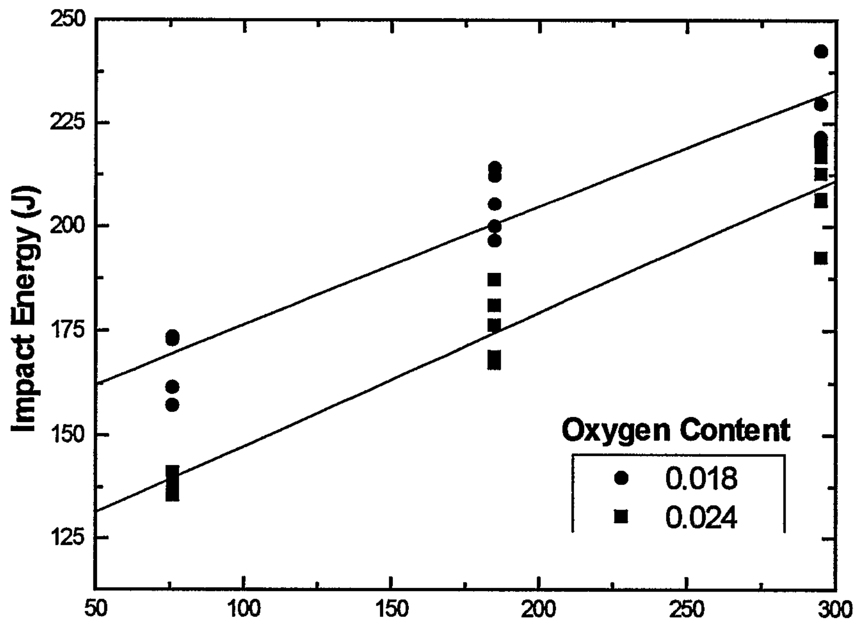

Figure 7
76 K CVN Impact Energy

The plot shows impact energy is directly affected by oxygen content and inclusion density, with that effect consistent throughout the temperature range tested. Also notable is the increase in data scatter at warmer temperatures.

Lateral expansion varied inversely with oxygen content and inclusion density, but consistently decreased with temperature (Figure 8). Lateral expansion ranged from 1.15 mm to 1.64 mm at 76 K, and at room temperature from 1.46 mm to 2.58 mm. Lateral expansion decreased an average of 25% with a 25% reduction in oxygen content, or with a 36% reduction in inclusion density. The data in Figure 8 also shows there is increased scatter with increased oxygen content, and a sharper decline in lateral expansion at lower temperatures with increased oxygen content.

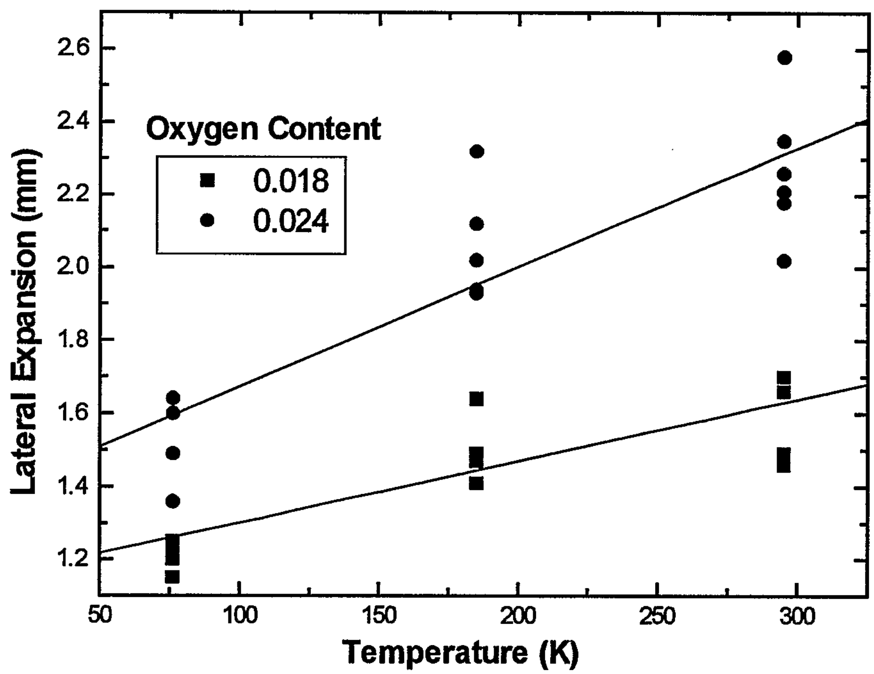

Figure 8
76 K CVN Lateral Expansion

The fractographic examinations revealed that all the welds failed in a ductile manner, with fracture surfaces exhibiting typical ductile dimple morphologies on a microscale, as shown by Figure 9 and Figure 10. It is also evident from these fractographs that the dimple size increases with increasing particle diameter. This observation is consistent with previous findings.[Refs. 23 and 24]

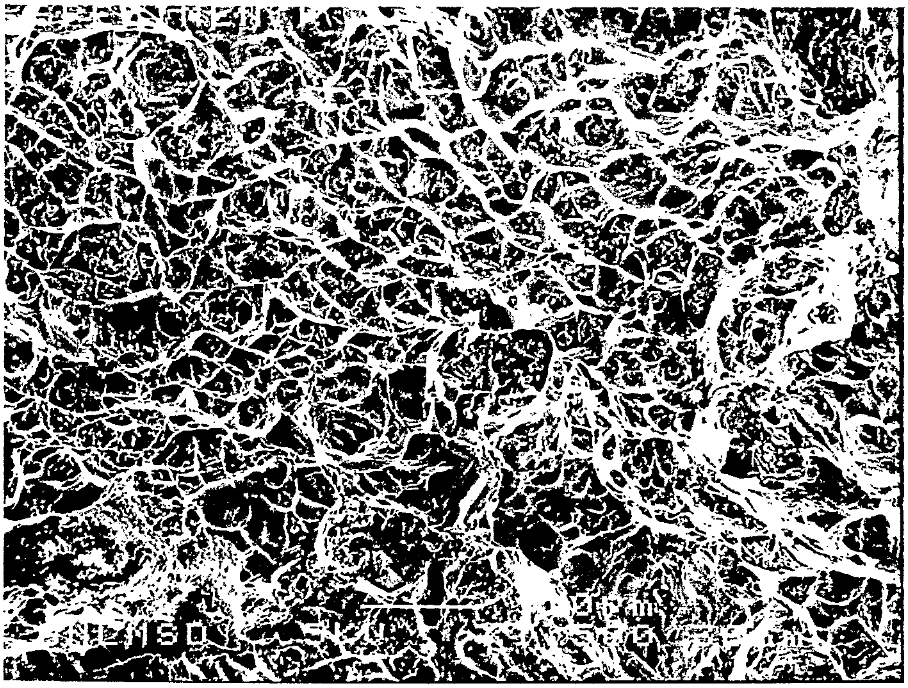

Figure 9
Fracture Surface of 76 K Charpy Specimen
(0.024% Oxygen Content) (1500 X)

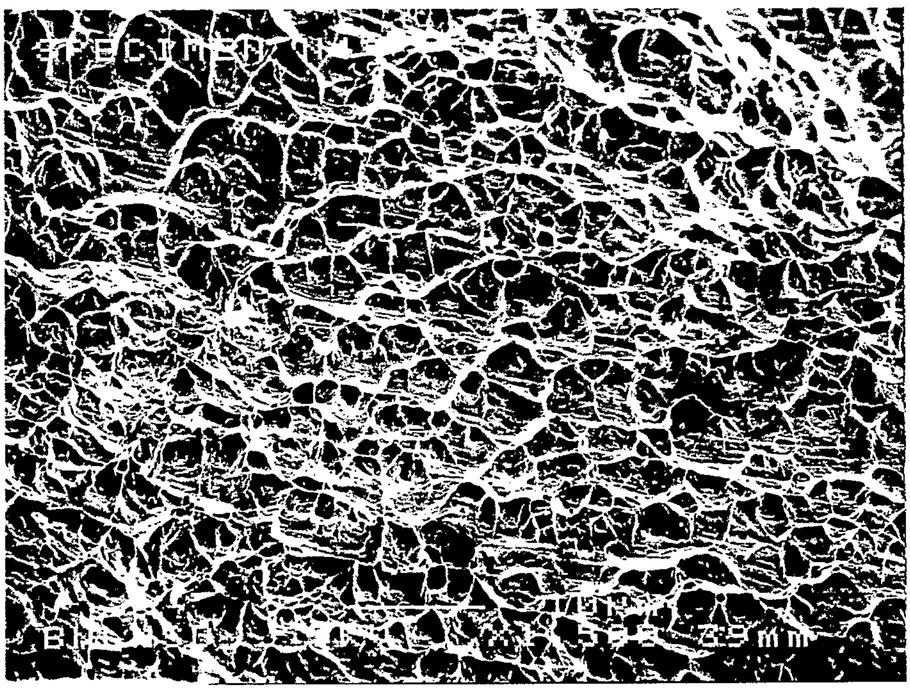

Figure 10
Fracture Surface of 76 K Charpy Specimen
(0.018% Oxygen Content) (1500 X)

Ferrite

The alloy is fully austenitic, that is, a material with a ferrite number less than 0.1. Using the WRC 1992, the tested alloys have a Nickel Equivalent of 29.5 (BNL Alloy) and 29.8, with a Chromium Equivalent of 26.2 to 26.5, yielding a ferrite potential of -5.6 to -5.4. Yet there have been no instances of hot cracking in over 1,000 kg of weld metal deposited to date. This can be attributed to the very low levels of phosphorous and sulfur, which were specified as a precautionary measure. Research (Ref. 13) has found that susceptibility to hot cracking exists when the sum of the weight percent of phosphorous and sulfur exceeds 0.01, but is almost eliminated with a Chromium Equivalent/Nickel Equivalent ($[Cr/Ni]_{eq}$) ratio greater than 1.48. In this study, the sum of the weight percent of phosphorous and sulfur is at least 0.015, but the $[Cr/Ni]_{eq}$ is 0.89, yet we see no cracking. It is possible that the manganese and copper additions beyond those of Reference 13 may be modifying the interdendritic regions and so reducing the cracking tendency.

Weldability

Weldability is a significant concern, especially in a production environment, and shielding gas has a major role. Manual gas tungsten arc welding with 100% argon had good welder appeal, bead appearance, and puddle control. Manual gas metal arc welding when using 100% argon scored poorly in welder appeal, bead appearance, puddle control, and wetting action. Automatic pulsed gas metal arc welding was impossible using 100% argon due to arc instability. All categories of weldability improved when 1% CO_2 was added to the shielding gas mixture, although further improvement in wetting action is desirable. Hydrogen and helium mixtures are recommended for this type of application, and preliminary testing showed marked improvement in GTAW weldability with the addition of hydrogen to the shielding gas.

CONCLUSIONS

This research leads to several conclusions.

1) A high nickel/high nitrogen superaustenitic weld alloy provides exceptional mechanical properties for a 4 K cryogenic welded structures using production welding processes. This is demonstrated in Figure 11, which shows this new alloy exceeds the minimum fracture toughness goal developed in the early 1980's.

2) Inclusion size distribution and inclusion density have a direct and significant effect upon weld metal fracture toughness. However, a procedure for determining inclusion density must be standardized before this can be used as a measure of fracture toughness.

3) Oxygen content has a direct effect on inclusion density, fracture toughness, and Charpy V-Notch energy. This is dependent upon the welding process, specifically, the ability of the process to exclude oxygen from the weld pool.

4) Hot cracking has not been experienced with this superaustenitic weld alloy. This may be attributed to the low phosphorous and sulfur content of the test alloys, but hot cracking susceptibility for stainless steels with $[Cr/Ni]_{eq}$ ratios less than 1.0 need additional investigation.

5) Adequate weldability was achieved with the addition of 1% CO_2 to the shielding gas, and did not adversely affect cryogenic material properties.

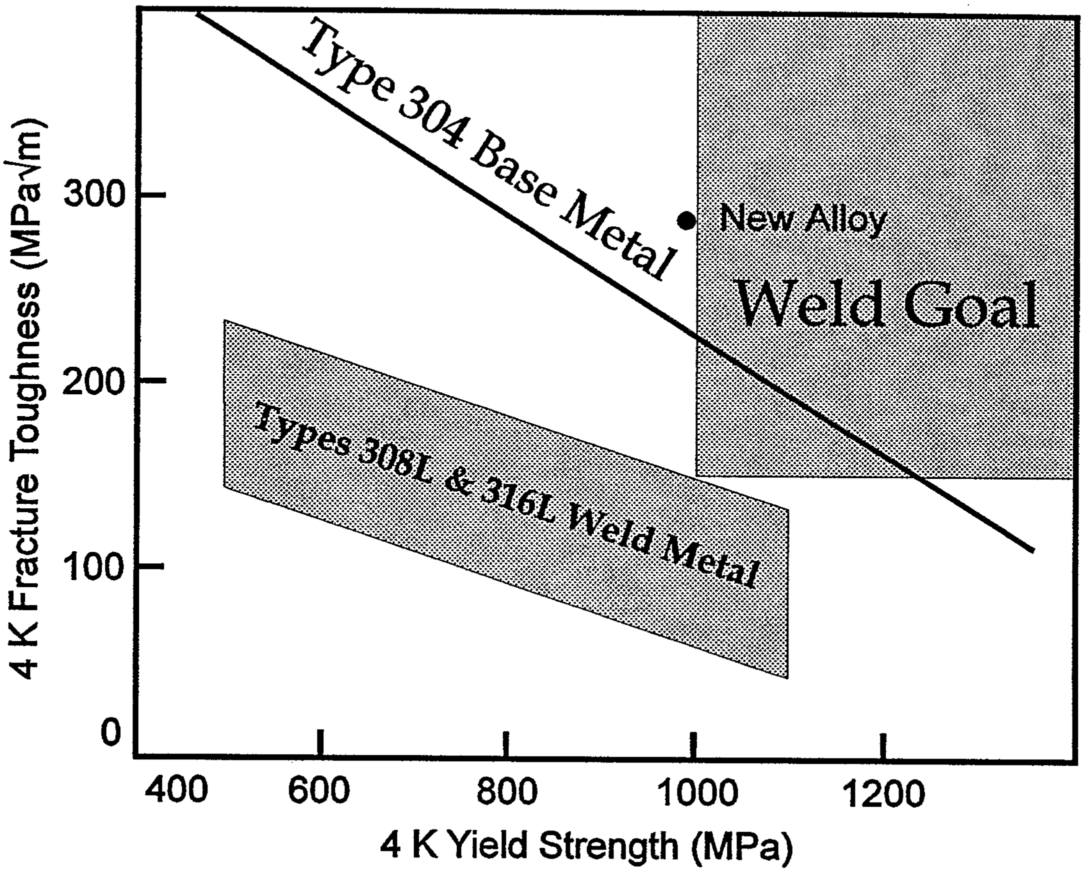

Figure 11
Test Alloy Mechanical Properties

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1 Tobler, R. L., Reed, R.P., Hwang, J.S., Morra, M.M., Ballinger, R. G., Nakajima, H., and Shimano, S. 1991. Charpy Impact Tests Near Absolute Zero, *Journal of Testing and Evaluation* 19(1): 34.

2 Tobler, R.L., Siewert, T.A., and McHenry, H.I. 1986. Strength-Toughness Relationship of Austenitic Stainless Steel Welds at 4 K, *Cryogenics* 26: 392.

3 Goodwin, G. 1985. Welding Process Selection for Fabrication of a Superconducting Magnet Structure, *Welding Journal* 64(8): 19.

4 Dalder, E.N.C., Seth, O.W., Whipple, T.A. 1981. Evaluation of Shielded Metal Arc and Flux Cored Metal Arc Welding Consumables for Fabrication of Stainless Steel Magnet Cases for 4 K Service, International Cryogenic Materials Conference, San Diego, CA; August 10-14, 1981

5 Reed, D.T., McHenry, H.I., Steinmeyer, P.A., and Thomas, Jr., R.D. 1980. Metallurgical Factors Affecting the Toughness of 316L SMA Weldments at Cryogenic Temperatures, *Welding Journal* 59(4): 104-s

6 Kim, J.H.,Oh, B.W., Youn, J.G., Bahng, G., and Lee, H. 1990. Effect of Oxygen Content on Cryogenic Toughness of Austenitic Stainless Steel Weld Metal, *Advances in Cryogenic Engineering (Materials),* Vol. 36B: 1339.

7 Siewert, T.A. and McCowan, C.N. 1992. Joining of Austenitic Stainless Steels for Cryogenic Applications, *Advances in Cryogenic Engineering (Materials)* 38B: 109.

8 McCowan, C.N., Siewert, T.A. Reed, R.P., and Lake, F.B. 1987. Manganese and Nitrogen in Stainless Steel SMA Welds for Cryogenic Service, *Welding Journal* 66(3): 84-s

9 Siewert, T.A. 1978. How to Predict Impact Energy from Stainless Steel Weld Composition, *Welding Design Fabrication* (June): 88

10 Szumachowski, E.R. and Reid, H.F. 1978. Cryogenic Toughness of SMA Austenitic Stainless Steel Weld Metals: Part I - Role of Ferrite, *Welding Journal* 57: 325-s

11 Szumachowski, E.R. and Reid, H.F. 1979. Cryogenic Toughness of SMA Austenitic Stainless Steel Weld Metals: Part II - Role of Nitrogen, *Welding Journal* 58: 34-s

12 Siewert, T.A. and McCowan, C.N. 1990. Cryogenic Mechanical Property Data for 20Cr-25Ni-4.5Mo Gas Metal Arc Welds, Materials Studies for Magnetic Fusion Energy Applications at Low Temperatures - XIII, NISTIR 3944, National Institute of Standards and Technology, 233

13 Kujanpaa, V., Suutala, N., Takalo, T., and Moisio, T. 1979. Correlation Between Solidification Cracking and Microstructure in Austenitic and Austenitic-Ferritic Stainless Steel Welds, *Welding Research International*, 9(2): 55

14 Simon, N.J. and Reed, R.P. 1986. Strength and Toughness of AISI 304 and 316 at 4 K, Materials Studies for Magnetic Fusion Energy Applications at Low Temperatures - IX, NBSIR 86-3050, National Bureau of Standards, Gaithersburg, MD: 27

15 McCowan, C.N. and Siewert, T.A. 1988. Inclusions and Fracture Toughness in Stainless Steel Welds at 4 K, *Advances in Cryogenic Engineering (Materials)*, Vol. 34B: 335

16 ASME Boiler & Pressure Vessel Code, Section II, SFA 5.4, A8, and SFA 5.9, A6

17 Whipple, T.A. and Kotecki, D.J. 1981. Weld Process Study for 316L Stainless Steel Weld Metal for Liquid Helium Service, Materials Studies for Magnetic Fusion Energy Applications at Low Temperatures - IV, NBSIR 81-1645, National Bureau of Standards, Boulder, CO 303.

18 McCowan, C.N. and Siewert, T.A. 1990. Fracture Toughness of 316L Stainless Steel Welds with Varying Inclusion Contents at 4 K, *Advances in Cryogenic Engineering (Materials)*, Vol. 36B: 1331

19 Brookhaven National Laboratory. February 16, 1993. Weld Filler Metal for RHIC Magnets, *RHIC Magnet Division Specification, RHIC-MAG-M-4360*

20 Siewert, T.A., Gorni, D. and Kohn, G. 1988. High-Energy-Beam Welding of type 316LN Stainless Steel for Cryogenic Applications, *Advances in Cryogenic Engineering (Materials)*, Vol. 34B: 343

21 Tobler, R.L., Read, D.T., and Reed, R.P., 1981. Strength/Toughness Relationship for Interstitially Strengthened AISI 304 Stainless Steels at 4 K, *Fracture Mechanics: 13th Conference*, ASTM STP 743, ASTM, Philadelphia, p. 250.

22 Kluken, A.O., Grong, Ø., and Hjelen, J. 1988. SEM Based Automatic Image Analysis of Non-Metallic Inclusions in Steel Weld Metals, *Materials Science and Technology*, Vol. 4, No.7: 649

23 Kluken, A.O., McCowan, C.N., and Siewert, T.A. 1993. Cryogenic Toughness of Austenitic Stainless Steel Weld Metals: Effect of Inclusions, Microstructural Science 20: ASM International, for the International Metallographic Society, Materials Park, Ohio, 45

24 Siewert, T.A. and McCowan, C.N. 1987. Welding Metallurgy of Structural Steels. Proceedings International TMS/EWI Symposium, Denver, 22-26 February, 1987. Ed.; J.Y. Koo. Publisher; Warrendale, Pennsylvania, 415