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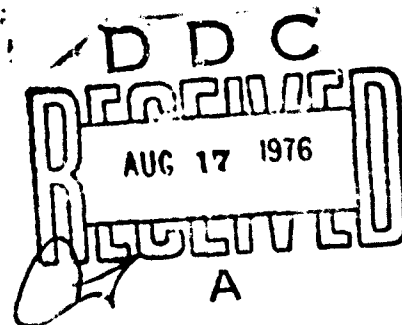
HYDROGEN IN HY-130 WELD METAL

July 31, 1976

by

D. G. Howden, E. G. Smith and R. M. Evans

 **Battelle**
COLUMBUS LABORATORIES
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) This report summarizes progress in producing welding electrodes containing known amounts of misch metal for reducing hydrogen embrittlement in HY-130 steel welds. Two methods of filler metal fabrication were evaluated during the period covered.		

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△ In one method an effort was made to produce wire with a misch metal core by extruding, rolling, and wire drawing a composite billet. This effort was not successful because of cracking during rolling the extruded billet. Based on this experience alternate methods, procedures for making the desired wire without rolling were outlined.

A specially fabricated cored electrode wire containing rare earth silicides rather than misch metal was evaluated in welding tests. Several difficulties were noted. These are most importantly related to the silicon content of the resultant welds and their mechanical properties. Production of useful welds was hampered by weld cracking when rare earth contents over 0.05 percent were used. Addition techniques are, at least partially responsible for the inability to make welds with higher concentrations of rare earths. Mechanical test results indicated losses in weld ductility which may not be tolerable. The cause of this may be the silicon or the rare earths or both.

Continuing work involves production of electrode wires containing only misch metal in order to isolate their effect on weld metal properties. ↗

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INTRODUCTION

This is the second annual report on the study of "Hydrogen in HY-130 Weld Metal", Contract No. N00014-74-C-0407 and covers the period from July 1975 to July 1976.

Extensive precautions are presently required when gas metal arc welding high yield strength steels in order to obtain adequate mechanical properties. This is because hydrogen from any source can cause cracking in the weld metal and heat affected zone and when at levels insufficient to cause cracking can decrease weld ductility. The present program is directed toward elimination of the hydrogen embrittlement problem by utilizing additives in the welding electrodes which will render hydrogen innocuous by chemically combining it to form hydrides.

In previous work on this program* it was shown that the addition of getter materials (misch metal) to HY-130 steel would tie up the diffusible hydrogen. Misch metal contents in the range 0.1 to 0.2 percent in experimental steels were capable of gettering hydrogen in steel in arc melting atmospheres of up to 5 percent hydrogen. Also, specially made filler wires containing about 0.2 percent misch metal operated without significant effect on welding operations. Problems of considerable magnitude were shown to exist in the fabrication of welding wires containing enough misch metal to overcome arc losses and give the desired weld metal composition.

It was found that 0.2 percent rare earth steel could be fabricated into 1/16 inch wire only with difficulty. Steels with higher than 0.2 percent misch metal suffered the problem of hot shortness during fabrication.

One of the main objectives of current work is to determine the effects of various rare earth levels on the weldability and mechanical properties of HY-130 weld metal. Two problems were foreseen:

* Annual Report, ONR Report No. N00014-74-C-0407 (NR031-770), dated July 31, 1975.

- (1) A hot shortness problem leading to weld hot cracking-- this could be expected from the limited knowledge available on the iron-rare earth element phase characteristics and the problems discovered earlier in the program during the hot working of HY-130 steel filler wire with a high rare earth level.
- (2) Deterioration of weld metal mechanical properties caused by the intergranular rare-earth rich phase observed previously in the structures of HY-130 with high rare earth content.

Efforts during the recent work, therefore, have been directed at investigating methods of introducing rare earths to the weld metal and examining the effect of these additions on the mechanical properties of HY-130 weld metal.

Several possible approaches were considered as methods of introducing rare earth elements into the weld pool without the necessity for alloying with the filler metal. All these methods assumed that rare earth elements could be fed into the weld pool via an auxiliary cold wire while the GMA arc was operated on HY-130 filler wire. They are outlined below.

(1) Addition of a Second Wire Containing Misch Metal Core.

Present technology indicates that tubular wires can be made down to a 0.035 inch diameter by inserting metallic powder inside a metal sheath and drawing the tube into a wire form. Unfortunately, misch metal powder of fine mesh size is pyrophoric and extremely difficult to handle. Discussions were held with manufacturers concerning ways in which a misch metal containing tubular wire could be made on an experimental basis, but it was concluded that the misch metal handling problems were too great.

(2) Addition of a Second Wire Containing a Misch Metal Core.

Misch metal in wire form is somewhat less pyrophoric than the powdered form. The possibility of inserting a misch metal wire in the center of a steel tube was considered. According to manufacturers, some problems

were likely to be encountered during wire drawing of this tubular product due to the dissimilar working characteristics of a steel sheath and misch metal wire core. An uneven distribution of misch metal in the wire could be expected. This approach was not pursued.

(3) Fabrication of a Composite Extrusion Billet for Producing an Auxiliary Wire. It is possible that, by taking a pure iron ingot, drilling a hole in the center, and casting misch metal into the hole, a composite billet could be produced. After sealing the ends of this billet, it could be hydrostatically extruded at a suitable temperature to minimize interaction between the misch metal core and the iron billet. Once the wire has been extruded, problems could arise with the distribution of misch metal during subsequent drawing of the wire to size. However, since the steel tube produced by this operation would not have a longitudinal seam, this technique was given a greater chance of success than the misch metal - sheath approach.

(4) Addition of a Second Wire Containing Rare Earth Silicide. In order to avoid the problem of pyrophoricity of fine mesh, misch metal powders, rare earth elements are often added to molten steel in the form of a silicide. A manufacturer agreed to produce two experimental batches of tubular wire of 0.045 inch diameter containing 1 percent and 10 percent rare earth silicide.

As indicated, Methods (1) and (2) were dropped from further consideration leaving Methods (3) and (4) for study in the current work. The production of composite wire from hydrostatic extrusion of a cored billet was attempted at Battelle-Columbus and the rare earth silicide wire was purchased from Hobart Brothers. Prime effort was made on Method (3) since this approach would introduce metallic rare earth elements to the steel, while Method (4) would involve adding silicon to the weld metal. It was not known if the increased silicon content would affect the hydrogen gettering capabilities of the rare earth elements.

FABRICATION OF A COMPOSITE EXTRUSION
BILLET FOR PRODUCING AN AUXILIARY WIRE -
METHOD 3

Introduction

The most desirable filler metal for placing rare earths in the weld metal is one that contains pure misch metal. In this way all of the effects of the addition can be determined. Thus, it was decided to produce the composite wire in two widely different misch metal concentrations of 1 and 10 w/o. Several fabrication schemes were considered for producing the composite welding wire, but a combination of extrusion, rolling, and drawing were selected as being most practical. Composite billets consisting of misch metal cores and carbon steel sleeves first would be prepared for reduction to wire. Hydrostatic extrusion was chosen because the characteristics of the process offer the best possibility for uniformly deforming the misch metal and steel billet components, which have widely different strength levels. Rod stock produced from extrusion was further processed into wire by cold rolling and drawing with intermittent heat treatments. The sections that follow describe the experimental efforts to product the misch metal-steel wire by these methods and the results obtained.

Preparation of Billets

Ten billets were prepared for the first fabrication step, hydrostatic extrusion; eight of these had misch metal cores and carbon steel sleeves while the remaining two had no cores.

Materials

The carbon steel sleeve material was procured in the form of wrought hot-rolled bar stock, nominally 2.75 inch (70 mm) in diameter.

This material had an AISI Type 1020 composition, but was obtained specifically because of its low phosphorus and sulfur contents which were 0.007 and 0.022 w/o, respectively. The bar stock had a very fine-grain annealed structure and its hardness measured 145-160 VHN.

Misch metal was obtained as a commercial alloy, Ceralloy 100X*, in the form of hot-extruded rods, nominally 0.2 and 0.5 inch (5.1 and 12.7 mm) in diameter. The rare earth metal content of the Ceralloy 100X alloy totals 99.7 w/o and consists of 50-52 percent cerium, 18-27 percent lanthanum, 12-18 percent neodymium, and 4-6 percent praseodymium. The misch metal rods had a hard outer surface resulting from interaction with the atmosphere when the rods were hot-extruded. Their hardness was 38-45 VHN over most of their cross section, and 600-675 VHN in the reacted zones on the surface.

Machining and Assembly

The steel bar stock was machined to a diameter of 1.70 inch (43.18 mm) and then cut into ten 5.91 inch (150 mm) long pieces. One end of each piece was machined with a full 45-degree conical nose. The misch metal rods also were machined taking off only enough material to remove the reacted surface bands and product straight sections, which were then cut into 3.94 inch (100 mm) lengths. Centerline holes were drilled into 8 of the 10 steel pieces from their flat surface end.

The machined steel and misch metal components were degreased prior to placing the misch metal in the holes of the steel sleeves. Steel end caps were then circumferentially electron beam welded to the back end of the sleeves, to protect the cores from the subsequent processing environments. Because it was necessary to machine the misch metal rods to remove reaction zone layers and achieve straight sections (in particular the large size rods), the concentration of misch metal in the composite billets was 1.1 and 5.7 w/o rather than the planned values 1 and 10 w/o. The placement of the misch metal

* A product of the Ronson Metals Corporation, Newark, New Jersey.

within the billet was checked radiographically before extrusion.

Lubrication

The billets were coated with a graphite-base lubricant, Deltaforge 907*. This lubricant was applied by heating the billets to ~93 F (200 C) and dipping them into an aqueous solution diluted to three parts water and one part lubricant. Successive dips of the billet produced a continuous layer of lubricant after evaporation of the water.

Extrusion Trials

The billets were extruded using hydrostatic techniques in a 2.36 inch (60 mm) diameter container system installed in a 700 ton (7 MN) hydraulic press. Extrusion variables included billet temperatures of 68, 750, 930 and 1200 F (20, 400, 500, and 650 C), ratios of 4:1, 6:1, 8:1, and 12:1, and stem speeds of 0.3 and 1.3 inch/s (8.5 and 34 mm/s). Castor oil was used as a hydrostatic fluid in all the trials. The initial trials were made at the higher temperatures and ratios to produce the smallest size product for further working. However, these conditions produced nonuniform deformation in the composite billets. Ultimately, it was necessary to cold-extrude the billets at low ratios to produce satisfactory rod material for further processing. Because of the limited number of composite billets prepared, some billets were not successfully extruded in earlier trials but reused.

A typical elevated temperature trial was made by preheating the billet and die to 750 F (400 C), quickly transferring the billet to the container, introducing a premeasured volume of castor oil into the container, and extruding the billet at a constant preset speed. The same procedures, except for preheating, were used for the ambient temperature trials. The extruded products from the trials were visually

* A product of the Acheson Colloids Company, Port Huron, Michigan.

examined and sometimes radiographed to determine the condition of their cores. Metallographic techniques were used to evaluate the products from a few trials.

Rolling and Drawing Operations

The composite rods from the extrusion trials were further processed first by rolling and then by drawing. The processing schedule for both procedures consisted of three consecutive passes totalling 35 percent reduction in area followed by heat treatment. Heat treatment was accomplished in air furnaces for 30 to 60 minutes followed by air cooling. A relatively low temperature of 930 F (500 C) was used to avoid excessive interaction between the misch metal core and carbon steel sleeve. After each 35 percent reduction sequence and heat treatment, the rods and wire were pickled in HCl to remove any oxide scale, and then visually, radiographically, and metallographically examined for defects.

Typical production rolling procedures promoted the formation of fins because the deformation was severe and nonsymmetrical. When the fins were not completely removed after each pass, they were rolled into the surface of the rod. Modification of these procedures were made to minimize the formation of fins.

The rolling operation was continued until the rod diameter was small enough to be handled on the drawbench. The product size at which rolling was stopped varied from about 0.25 to 0.5 inch (6 mm to 12 mm) diameter, the largest size product that can be drawn with existing equipment. A commercial lubricant, Houghton Wire Draw 3105*, was used to draw the composite rods.

* A product of the E. F. Houghton and Company, Detroit, Michigan.

Results and Discussion

Extrusion

The results from the 15 extrusion trials made are summarized in Table 1. As mentioned earlier, it was necessary to reuse several billets not successfully extruded in earlier trials because of the limited number of billets prepared. The inability to extrude billets was due to excessively high pressure or to problems associated with the tooling itself. Since tooling problems were encountered in Trial Numbers 6, 7, and 8, and definitely affected the results, these trials can be ignored. In addition, the failure to extrude the reused billet in Trial Number 15 probably reflects work hardening effects in the nose section acquired from earlier extrusion attempts.

Elevated Temperature Trials. The early warm extrusion trials reflect an attempt to produce a product small enough to go straight into the drawing operation without the need for rolling. Based on available supplier data for the Ceralloy 100X alloy, melting should not occur below about 1500 F (815 C). Therefore, an extrusion temperature of 1200 F (650 C) was selected for the first series of warm trials.

Extrusion at 1200 F (650 C) and a 12:1 ratio produced composite rods (Trial Numbers 1, 2, and 3) with periodic enlarged regions along their length. Similar results were obtained when extruding at 930 F (500 C) and a 12:1 ratio (Trial Number 4) except the enlarged regions were smaller. Radiographs clearly showed periodic buildups of misch metal corresponding to the enlarged regions of the rod. Regions between these packets, in contrast, showed only a thin line of core material. These results indicate that nonuniform deformation of the core and sleeve took place due to large differences in flow stress at the extrusion temperatures used. The misch metal core being much softer than the steel sleeve deformed preferentially and was trapped periodically in discrete

TABLE 1. SUMMARY OF HYDROSTATIC EXTRUSION TRIALS FOR COMPOSITE BILLETS

Trial Number	Billet Number	Billet Comp., % Misch metal	Billet Temp. F (C)	Nominal Extrusion		Extrusion Pressure, ksi (MPa)	Product Dimensions	
				Ratio	Breakthrough		Runout	Diameter, in. (mm)
<u>Elevated Temperature Trials</u>								
1	1	0	1200 (650)	12:1	1300	1080	0.48 (12.3)	4.99 (1.52)
2	2	5.7	"	"	1350	1050	0.48 (12.3)	5.84 (1.78)
3	3	1.1	"	"	1260	1050	0.48 (12.3)	5.90 (1.80)
4	4	1.1	930 (500)	"	1660	1410	0.48 (12.3)	4.76 (1.45)
5	5	1.1	750 (400)	"	1790 (a)	--	--	--
6	6	0	"	8:1	1650 (a)	--	--	--
7	6 (b)	0	"	8:1	1690 (a)	--	--	--
8	5 (b)	0	"	6:1	1500 (a)	--	--	--
<u>Ambient Temperature Trials</u>								
9	6 (b)	0	68 (20)	4:1	1150	1100	0.86 (21.9)	1.67 (0.51)
10	7	1.1	68 (20)	4:1	1100	1100	0.86 (21.9)	1.25 (0.38)
11	8	5.7	68 (20)	6:1	1400	1400	0.70 (17.9)	3.18 (0.97)
12	9	5.7	68 (20)	6:1	1420	1420	0.70 (17.9)	2.49 (0.76)
13	10	5.7	68 (20)	6:1	1400	1400	0.70 (17.9)	2.49 (0.76)
14	5 (b)	1.1	68 (20)	6:1	1460	(c)	0.70 (17.9)	0.59 (0.18)
15	5 (b)	1.1	68 (20)	6:1	1750 (a)	--	--	--

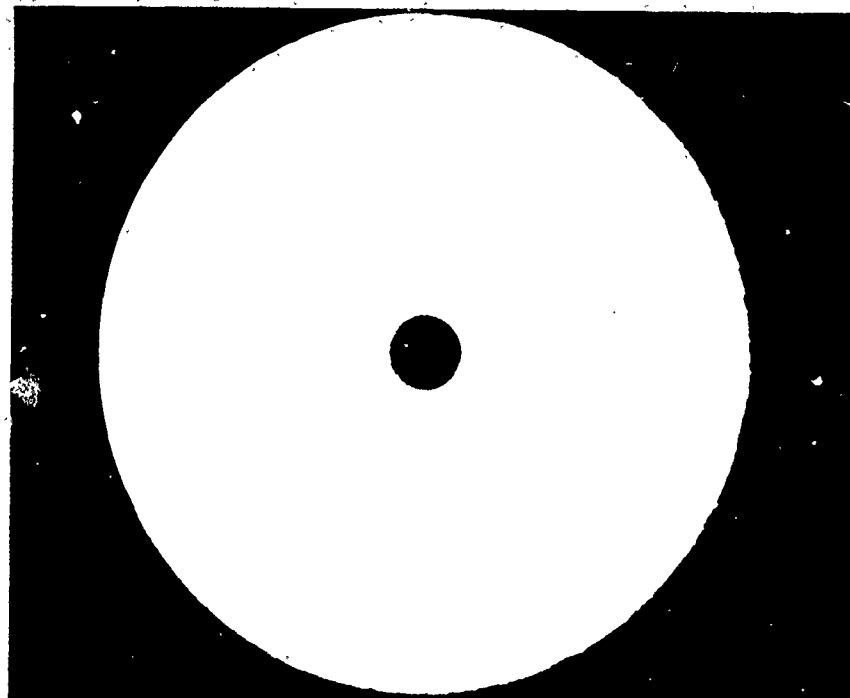
- (a) No breakthrough achieved - trial stopped because of high pressure or problem with tooling
 (b) Billet not extruded in earlier trial reused
 (c) Poor lubrication and work hardening produced severe stick-slip condition and trial was terminated early.

packets. Metallographic examination further revealed that partial melting of the misch metal cores had taken place in all of the warm trials. Evidently the deformation heat resulting from extrusion raised the core temperature close to or above 1500 F (815 C), the reported solidus temperature for the Ceralloy 100X alloy.

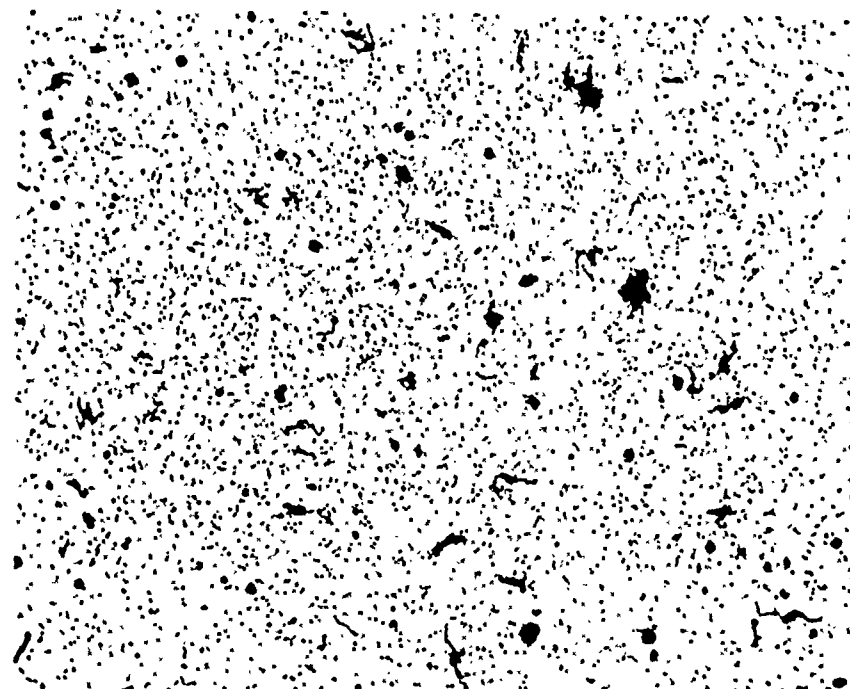
The results of the previous trials indicate that the lower billet temperatures would promote more balanced core-sleeve flow in the composite billets. Therefore, several additional trials were made using a 750 F (400 C) billet temperature and extrusion ratios of 8:1 and 12:1 (Trial Numbers 5, 6, and 7). Excessively high pressures prevented extrusion in these trials. The same billet was used in all these trials and the extraneous effect of work-hardening in the nose section may have contributed to these results. Extrusion at 750 C (400 C) and a 6:1 ratio (Trial Number 8) was also unsuccessful, but tooling problems definitely affected the results of this trial.

Ambient Temperature Trials. The results of the warm trials indicated that the pressure requirements for cold extrusion would not be much greater than those for extrusion at 750 F (400 C) and the same ratio. In addition, flow stress in the misch metal core and steel sleeve would be better matched at ambient temperature and billets should deform more uniformly.

Cold extrusion of the remaining composite billets was successfully accomplished first at a 4:1 ratio (Trial Numbers 9 and 10) and then at a 6:1 ratio (Trial Numbers 11, 12, 13, 14, and 15). The extruded rods produced had excellent surface finishes and uniform external dimensions. Radiographs were taken of these rods to show the relative dimensions of core and sleeve. The uniformity of the core-sleeve deformation produced in these rods is illustrated in the macrographs of Figures 1(a) and 2(a). Measurements of the cores and sleeve indicate that the cores in these rods reduced in the same ratio as the sleeves, so as to maintain their original core concentrations of 1.1 and 5.7 w/o, respectively. The microstructures of misch metal cores in two cold-extruded rods are shown in Figures 1(b) and 2(b).



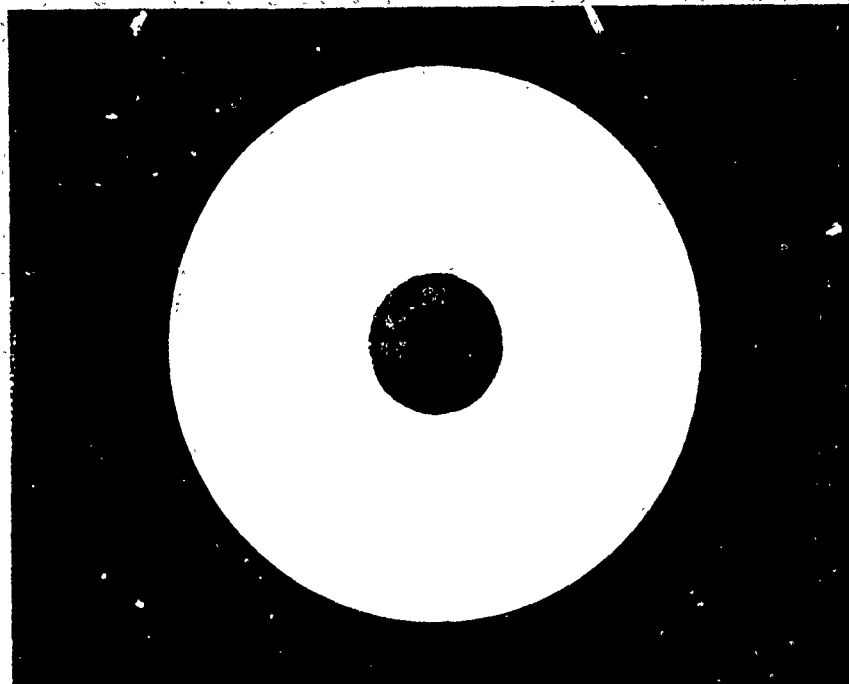
(a) 4X Core Diameter: 0.095 in. (2.41 mm) 8H441
Sleeve Diameter: 0.862 in. (21.9 mm)



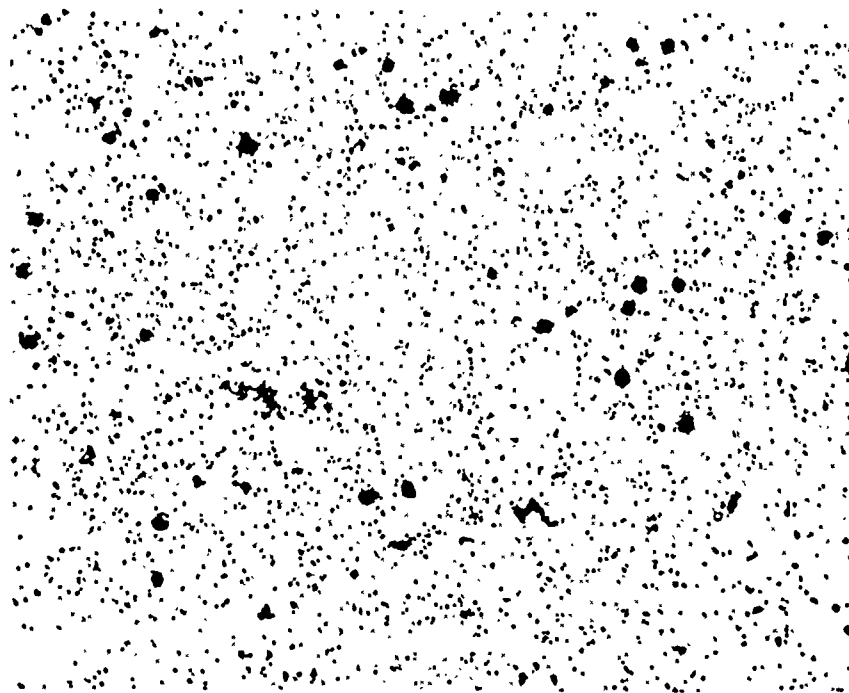
(b) 250X As-polished 8H439
Core Hardness: 55-61 VHN

FIGURE 1. BILLET NO. 7 AFTER COLD EXTRUSION AT 4:1

The as-extruded material shows no cracks and the relative core-sleeve dimensions indicate the nominal 1:1 w/o core composition of the starting billet has been maintained during extrusion.



(a) 4X Core Diameter: 0.181 in. (4.60 mm) 8H442
Sleeve Diameter: 0.704 in. (17.9 mm)



(b) 250X As-Polished 8H439
Core Hardness: 55-68VHN

FIGURE 2. BILLET NO. 10 AFTER COLD EXTRUSION AT 6:1

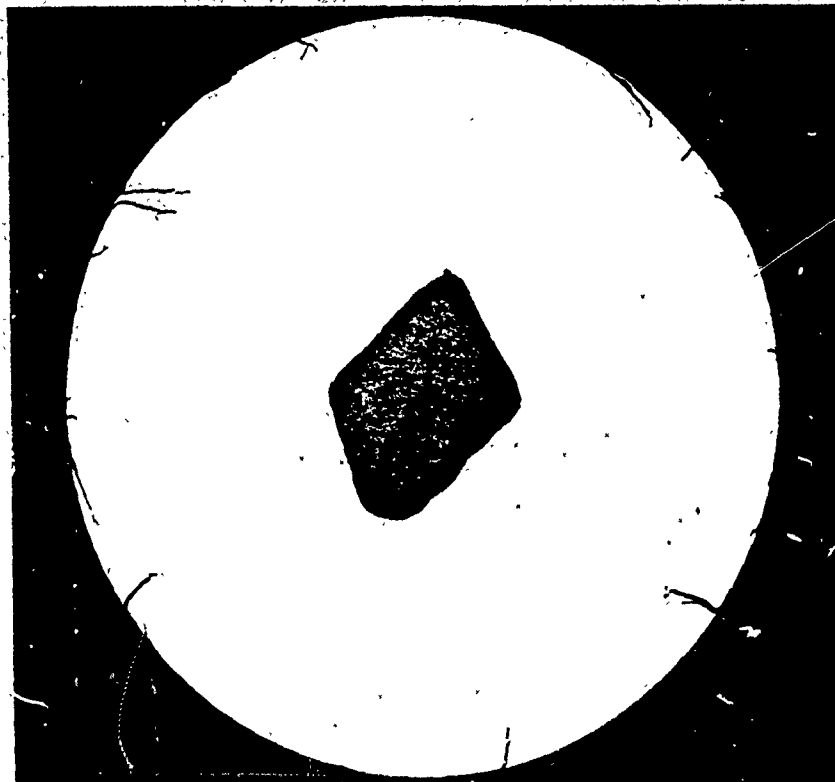
The as-extruded material shows no cracks and the relative core-sleeve dimensions indicate that the nominal 5.7 w/o core composition of the starting billet has been maintained during extrusion.

Several lengths of composite rod with 1.1 and 5.7 w/o misch metal cores were successfully produced by cold extrusion. These rods were used in subsequent rolling and drawing studies to produce the target-size composite wire.

Rolling and Drawing

The composite rods successfully cold-extruded were heat treated and further cold reduced using a combination of rolling and drawing operations. In the first series of experiments, the as-extruded rods were rolled to a nominal diameter of 0.24 inch (6.0 mm) before drawing was started. The wires produced from these efforts exhibited nonround cores and numerous surface cracks. A typical wire from this series, shown in Figure 3, was reduced from 0.70 to 0.18 inch (17.9 to 4.63 mm) diameter. The 5.7 w/o core in this wire has a diamond shape. Further drawing of this wire produced continuous longitudinal splits as the cracks propagated through the sleeve to the core.

The nonround cores and surface cracks produced in the composite wires from the first series of experiments are attributed to effects from the rolling operation. It is believed that the first few passes through the rod rolls were too severe for the relatively soft misch metal cores, and the cores were deformed out-of-round at an early stage of rolling. The deformed core shapes were then maintained through later stages of drawing. Nonuniform deformation associated with the early stages of rolling also produced fins on the surface of the rods. Although such fins are not uncommon in rod rolling, it is beneficial to remove them between passes to avoid their being rolled into the surface of the rods during subsequent rolling operations. It appears that fins produced from the severe deformation of the early stages of rolling were not adequately removed after each pass, and thus were rolled into the rod surface and served as nucleation sites for cracks which developed at later stages of the rolling and drawing sequence.



(a) 20X Sleeve Diameter: 0.19 in. (4.83 mm) 8H488



(b) 250X As-Polished 8H449

FIGURE 3. COMPOSITE WIRE FROM FIRST SERIES OF ROLLING AND DRAWING EXPERIMENTS

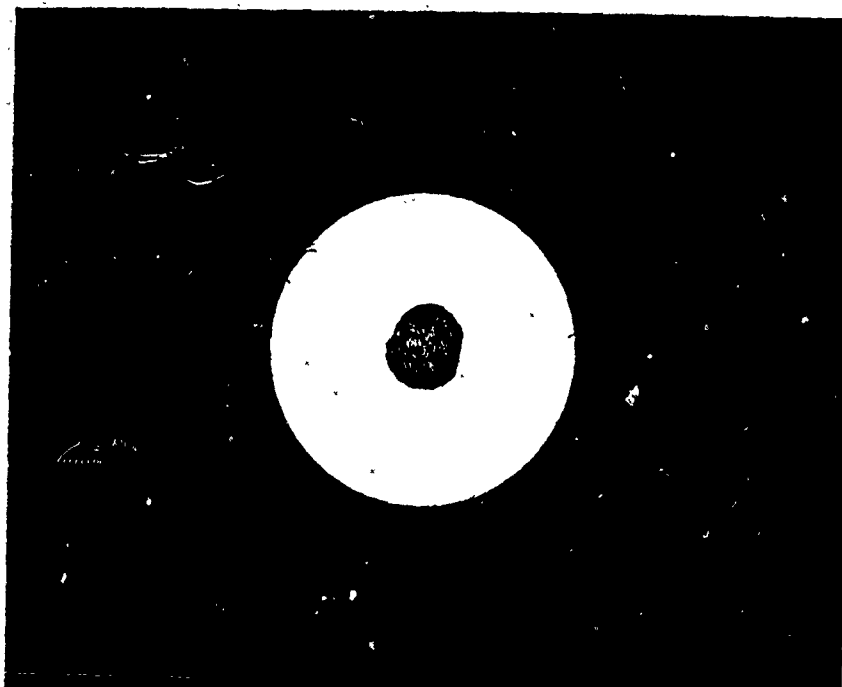
The non-uniform core shape and surface cracks were typical features in the wires produced in the early experiments.

A second series of extruded rods were cold-rolled during which precautions were taken to deform the rods as uniformly as possible through each rolling pass and minimize the formation of fins. Any fins produced were removed as completely as possible to avoid their being rolled into the rod surface. Each rod was rolled only until its diameter was about 0.47 inch (12 mm) at which stage it was further reduced by drawing.

The composite wires from these experiments were reduced to a smaller diameter than in the earlier work before longitudinal seams opened up along their length. Also, these wires maintained their round cores and had fewer surface cracks, as illustrated by the two wires shown in Figures 4 and 5. The top section in each figure was taken from a sound region in the wire, and the lower sections were taken from regions where seams had opened up. Differences in core structure are due to exposure to air during heat treat cycles at 932 F (500 C). In Figure 4 (lower) the core has completely reacted to produce a complex ceramic having a hardness of 650 VHN. The core of the wire in Figure 5 has only partially reacted and shows a sharp difference in hardness from the unreacted zone (43-45 VHN) to the reacted zone (680 VHN). Evidently the longitudinal seams had already opened up when these wires were given their last heat treat cycle.

The cracks as exhibited in Figures 4 and 5 appear to have initiated from the same source as those discussed earlier, even though careful rolling procedures were used to avoid fins.

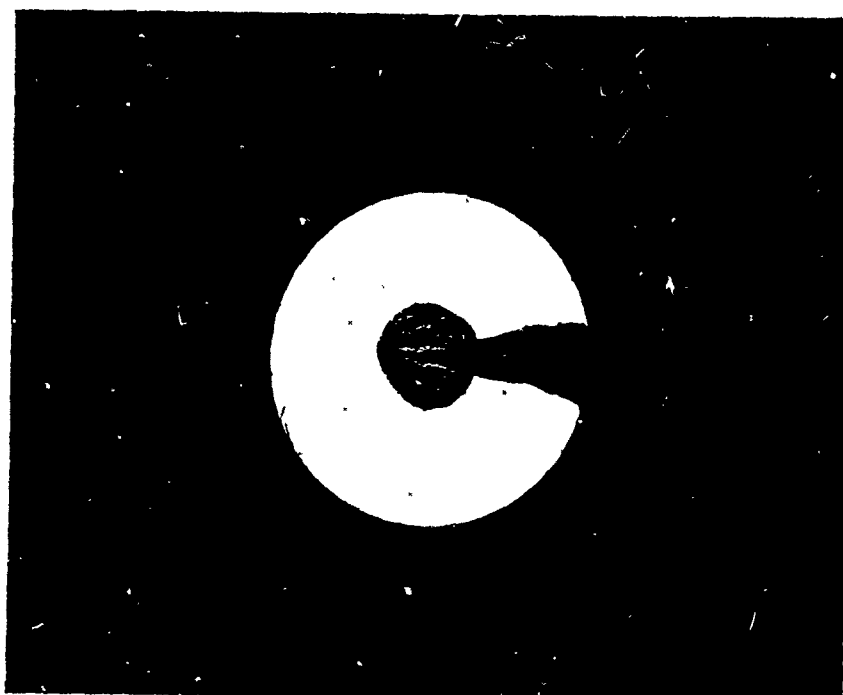
Another factor that may have contributed to the propagation of cracks in the composite wires is the low temperature used in the heat treat cycle. A temperature of only 932 F (500 C) was used to avoid any reaction between the core and sleeve components that might produce brittle reaction products and adversely affect the ductility of the composite wire. Although the 932 F (500 C) temperature is sufficient to stress-relieve the carbon steel sleeve, it may not fully remove all the prior cold working effects. It is possible that an accumulation of cold work during the rolling and drawing operations reduced the ductility



(a) 20X

Core Diameter: 0.021 in. (0.53 mm)
Core Hardness: 44 VHN

8H446



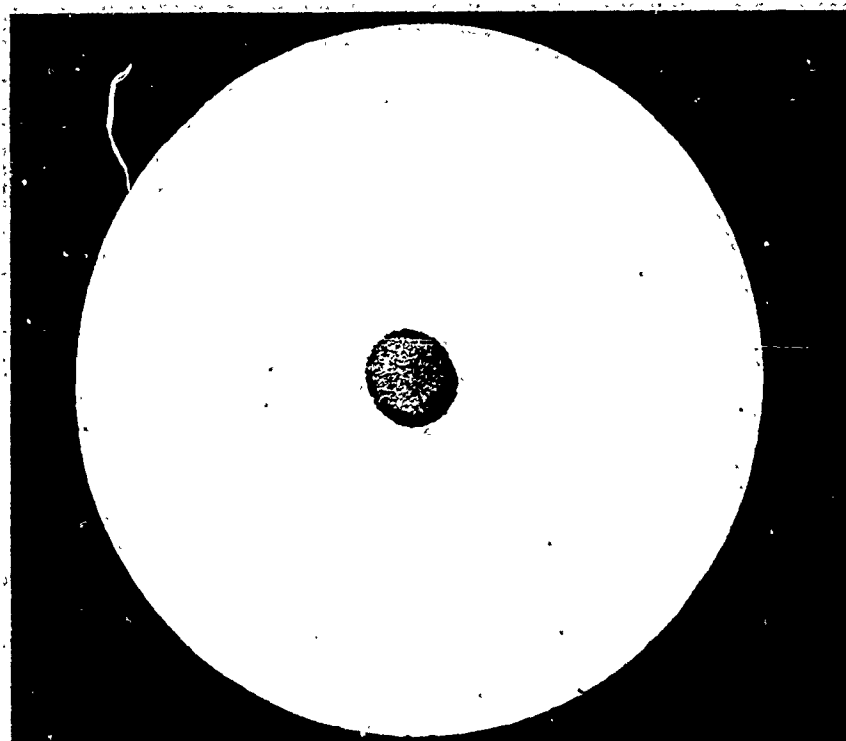
(b) 20X

Core Diameter: 0.021 in. (0.53 mm)
Core Hardness: 650 VHN

8H447

FIGURE 4. MACROSCOPIC SECTIONS THROUGH COMPOSITE WIRE
WITH 5.7 PERCENT MISCHMETAL CORE

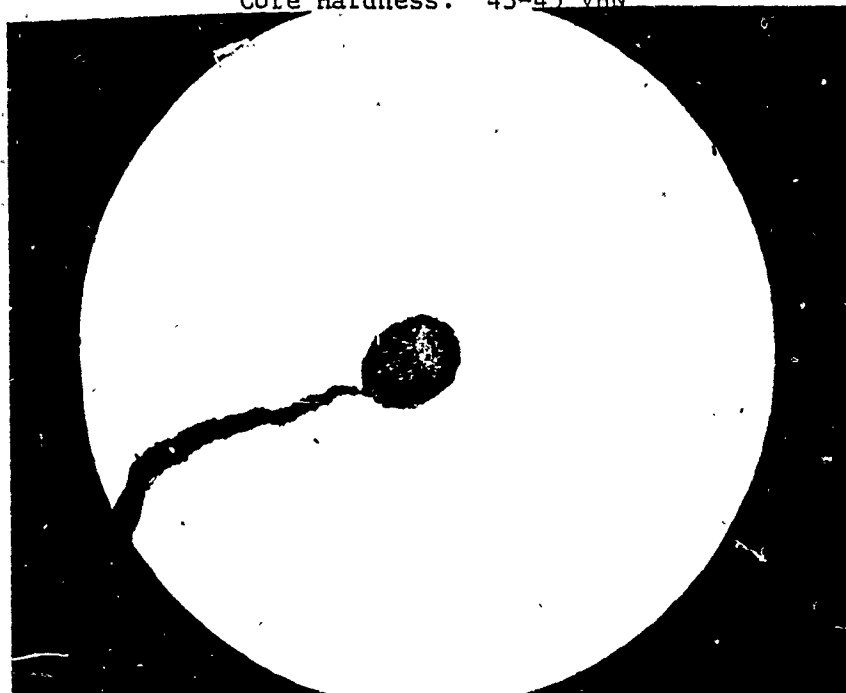
Shown are sound and cracked regions from a
wire after cold reducing to 0.088 in. (2.23 mm)
diameter and heat treated at 932 F (500 C)



(a) 20X

Core Diameter: 0.025 in. (0.64 mm)
 Core Hardness: 43-45 VHN

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(b) 20X

Core Diameter: 0.025 in. (0.64 mm)
 Core Hardness: 43-45 VHN (unreacted zone)
 680 VHN (reacted zone)

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FIGURE 5. MACROSCOPIC SECTIONS THROUGH COMPOSITE WIRE WITH 1.1 PERCENT MISCHMETAL CORE

Sections are shown through sound and cracked regions from wire cold reduced to 0.182 in. (4.63 mm) diameter and heat treated at 932 F (500 C)

of the sleeve material such that cracks once formed could propagate more easily through the sleeve.

Extrusion and Rolling Study Conclusions

The results of this fabrication study to produce misch metal steel composite welding wire of 0.040 inch (1.0 mm) diameter indicate the following conclusions.

- (1) Cold hydrostatic extrusion promotes uniform deformation in composite billets having a misch metal core and carbon steel sleeve.
- (2) Nonsymmetrical deformation of composite rods during cold rolling produces fins which are rolled into the rod surface.
- (3) Surface cracks believed to initiate at the rolled-in fins propagate through the steel sleeve during subsequent processing, and result in continuous longitudinal splits in the wire.
- (4) The heat treat temperature of 932 F (500 C) used in the processing sequence is not high enough to remove all cold work from the steel sleeve, and may contribute to crack propagation in this component.

Recommendations for Further Work on Extrusion and Rolling of Composite Wire

The successful fabrication of misch metal-steel composite welding wire was not realized during this program because of cracks that developed in the wire during the rolling and drawing operations. These cracks are believed to initiate from fins formed and rolled into the surface during the rolling operation. The cracks thus formed at the surface propagated through the steel sleeve during subsequent drawing

operations, resulting in wire with continuous longitudinal splits. A second contributing factor was the low temperature, 932 F (500 C), used in the heat treat cycle, which was not high enough to remove prior cold work in the steel, thus making it more susceptible to crack propagation.

If the rolling operation were eliminated from the fabrication sequence and the heat treat temperature increased to 1100-1200 F (600 to 650 C), the possibilities of producing the target-size composite wire would very much be improved. Elimination of the rolling operation can be realized in practice by extruding a rod product sufficiently small that it can be handled on the drawbench. Available draw dies could accommodate a 0.47 inch (12 mm) diameter rod. The same drawing schedule, that is, three consecutive 10 percent area reduction passes followed by heat treatment, would be used to produce the 0.040 inch (1.0 mm) diameter target-size wire.

ADDITION OF A SECOND WIRE CONTAINING RARE
EARTH SILICIDE: METHOD 4

As indicated previously, experimental electrode wires containing rare earth silicide were produced especially for this program. The major effort covered by this section of the report is the fabrication and testing of weldments made while adding wires containing rare earth silicides.

Weldment Preparation

Test Specimens

The weldment utilized to produce all-weldmetal specimens for tensile strength and impact property determinations is based on that specified in MI-30-BE/1, "Procurement Specification Electrodes, Welding, Bare, Solid, Manganese-Nickel-Chromium, Molybdenum Alloy Steel for Producing HY-130 Weldments for As-Welded Applications". Modification was necessary to minimize the amount of electrode wire expended. The root gap was reduced to 1/8 inch (3.18 mm) and a standard 3/8 inch (9.53 mm) tension test specimen was used in place of the 1/2 inch (12.7 mm) specified, impact test specimens were of standard CVN design.

Materials

The base metal being used for producing weldments is 1 inch thick (25.4 mm) HY-140 grade steel plate having the following analysis.

Nickel	4.76 percent	Vanadium	0.076 percent
Manganese	0.84 percent	Copper	0.060 percent
Chromium	0.49 percent	Aluminum	0.030 percent
Molybdenum	0.36 percent	Carbon (total)	0.120 percent
Silicon	0.34 percent	Phosphorus	0.003 percent
Zirconium	0.11 percent	Sulfur	0.007 percent.

The primary electrode wire for welding was furnished by the Sponsor. It is an experimental wire known as AX-140 and is 1/16 inch (1.59 mm) in diameter, bare and bright. It was produced by Airco Welding Products and is recorded as Heat Number 51252 Lot LK4.

The rare earth containing cored wire was fabricated to 0.045 inch (1.14 mm) diameter by Hobart Brothers. This wire was made by placing rare earth silicide powder inside a low carbon steel sheath and wire and drawing to size. The nominal compositions of the rare earth silicide powder used was 33 percent rare earth, 35 percent silicon and 12 percent iron. Two lots of wire were fabricated as given here:

<u>Lot No.</u>	<u>Wgt. Percent Wire Fill</u>	<u>Wgt. Percent Rare Earths</u>	<u>Wgt. Percent Cerium in core</u>	<u>Wgt. Percent Silicon in core</u>
392-6B	10.76	3.55	1.78	3.77
392-6A	0.91	0.30	0.15	0.32

Only Lot 392-6B is being used to produce welds because it permits minimum addition of the cold wire.

Welding Procedures

The method used to place cerium (rare earths) in the weld was to cold feed a cored wire containing the additive into the forward edge of the gas metal arc weld pool. The welding parameters were set suitable for normal operation using 1/16 inch (1.59 mm) diameter electrode wire. The 0.045 inch (1.14 mm) diameter cored wire was fed into the weld pool at a rate suitable to overcome arc losses and reach the desired cerium concentration level. This rate was calculated from data obtained by the analysis of bead-on-plate welds when using cored wire containing 10.76 percent misch metal and a primary electrode feed rate of 194 ipm (4.93 mpm). The results of these bead-on-plate studies are given in Table 2.

TABLE 2. RECOVERY EFFICIENCY OF RARE EARTH ADDITIONS

Pad No.	Shielding Gas	Cold Wire Feed Rate, ipm (mm)	Calculated Desired Rare Earth Content %	Analyzed Cerium Content, %	Estimated Actual Rare Earth Content, %	Rare Earth Recovery Efficiency %
1	Argon	20 (508)	0.10	0.017	0.034	34
2	Argon + 2% O ₂	20 (508)	0.10	0.011	0.022	22
3	Argon	62 (1575)	0.10	0.051	0.102	--

The welding parameters utilized gave a range of heat inputs between 45,000 and 47,000 joules per inch (17,700 to 18,500 joules per cm) when using a travel speed of 10.5 ipm (2.67 mm). The electrode wire feed rate was 194 ipm (4.93 mm). Either pure argon or argon plus 2 percent oxygen flowing at 40 cfh (1.13 cmh) was used for shielding the arc. The base plate was preheated to 250 F (120 C) before welding began and maintained between 200 and 250 F (93 and 1120 C).

Welding Results

The objective is to produce specimens of the described weldment having three levels of rare earth concentration in the weld metal (0.05, 0.10 and 0.20 percent) for the determination of weld metal mechanical properties.

To the present only welds containing an intended 0.05 percent rare earths have been made successfully. Centerline cracking of the early weld bead passes has prevented completion of welds with higher rare earth concentrations. Variations in welding parameters within practical limits to give lower and higher heat inputs has not aided in crack elimination. Another possibility, deposition of small weld beads, has not yet been tried.

The addition of a second wire containing rare earth silicides to the weld metal presents problems believed to be related to the silicon

Silicon in Weld Metal. Previous work has shown that from 0.1 to 0.2 percent rare earths are desirable for tying up the diffusible hydrogen. Also experience indicates that rare earth recovery from the welding arc is only around 25 percent. Therefore, when additions are made using rare earth silicide wire containing only 3.55 percent rare earths and a similar amount of silicon it is necessary to add large quantities of the much more recoverable silicon at the same time the rare earths are added. Calculations show that when adding enough rare earth containing wire to reach 0.1 percent rare earths in the present studies enough silicon was added to cause the weld metal to contain about 0.80 percent silicon. This level of silicon in low alloy high-strength steels can be a source of difficulties such as reduced toughness and possibly increased tendency for hot cracking of the weld metal. Actual analyses of two different weld metals with an intended 0.05 percent rare earth showed 0.60 and 0.50 percent silicon. An incomplected weld with an intended 0.10 percent rare earth showed 0.62 percent silicon.

Hydrogen Gettering by Rare Earth Silicide Wire. It was mentioned in the report introduction that there is a possibility that silicon may affect the hydrogen gettinging capabilities of the rare earth elements from the silicide. Thus, hydrogen gettinging experiments were made on several melted down buttons of steel-rare earth silicide wire, and samples of ingots made during the original work on the program. A standardized procedure of melt down with a gas tungsten arc torch using a gas mixture of argon plus 2 percent hydrogen was used. Immediately after melt down buttons were quenched in water and then placed under an inverted funnel in hot glycerin at 150 F (66 C) to trap the hydrogen which evolved. The results of this effort are given in Table 3. The results show that the hydrogen gettinging capabilities of the rare earths are not greatly different whether or not they are introduced to the weld metal as a neat mixture or as a rare earth silicide.

TABLE 3. HYDROGEN GETTERING TESTS OF SEVERAL STEEL MATERIALS

Material Identification	Wgt. of Button, g	Total gas evolved, ml	Gas evolved per unit wgt., ml/g
Ingot section from prior work, 0.00 percent misch metal	14.0	0.45	0.032
Ingot section from prior work, 0.05 percent misch metal	9.0	0.12	0.013
Ingot section from prior work, 0.10 percent misch metal	12.0	0.16	0.013
Ingot section from prior work, 0.30 percent misch metal	9.0	0.05	0.005
Ingot section from prior work, 0.70 percent misch metal	12.0	0	0
One part rare earth silicide wire, one part AX140 wire	7.0	0	0
One part rare earth silicide wire, two part AX140 wire	12.0	0	0
One part rare earth silicide wire, three part AX140 wire	12.0	0	0
AX140 solid welding wire			
Wire from extrusion study, 5.7 percent misch metal			
Wire from extrusion study, 1.1 percent misch metal			

Mechanical Properties of Welds. A major concern during this program is the effect of weld metal additions on the mechanical properties of the weld metal. The addition of the rare earth and also any composition change caused by the form of rare earth additive may lead to the reduction of desirable mechanical properties. Therefore the successful welds made were subjected to standard tensile and notch toughness tests. One control weld specimen, containing no rare earth silicide, and two weld specimens having an intended 0.05 percent rare earth addition were tested. The results are given in Table 4. The data show a slight increase in tensile properties of the rare earth containing welds over the control weld. The ductility as measured by elongation is lowered on the same comparison. The notch toughness of the weld metal was significantly reduced by the addition of the rare earth silicide, and there is an indication that the transition temperature may be around 30 F.

The changes in mechanical properties are the same as would be expected by the addition of silicon. Since silicon was added to about the 0.60 percent level at the same time the rare earth was added, their individual effect is not apparent.

Based on present criteria it appears that the addition of rare earths to the weld pool from a composite wire which contains rare earth silicide will be impractical because of the undesirable influence on mechanical properties.

TABLE 4. MECHANICAL PROPERTIES OF EXPERIMENTAL WELDS

Spec. No.	Weld Metal	Shield Gas	Heat Input		Yield Strength*		Tensile Strength		Impact Strength			Elongation %, 1 inch
			J/in	J/cm	ksi	MPa	ksi	MPa	CVN**, ft/lbs			
									30° F			
									-1 C	25 C	77° F	
5	AX-140+RES	Argon+2% O ₂	46,000	18,100	131.9	92.8	155.9	109.6	42	53	23	
6	AX-140	Argon+2% O ₂	46,000	18,100	130.7	91.9	146.1	102.7	70	58	26	
7	AX-140+RES	Argon	47,000	18,500	138.7	97.5	150.2	105.6	44	53	24	
(a)	--	--	35,000	13,800	135.-	95.-	--	--	--	--	--	
			45,000	17,700	145.0	102.0	--	--	50	59	14***	

* Average of 2 specimens

** Average of 5 specimens each temperature

*** Elongation in 2 inches

(a) MI-30-BE/1 Requirements

Discussion of Welding Results

The production of satisfactory welds when adding rare earths from a cored cold wire containing rare earth silicides has been difficult. The difficulties appear to arise from two sources; mainly the presence of large concentrations of silicon in the wire but also from the procedures utilized to make the addition. Other techniques for adding the wire can be devised but it is felt that other features of the use of rare earth silicide should be considered before further effort is expended on the addition procedure.

Hydrogen gettering tests show that silicon does not reduce the potency of the rare earths present. But by calculation and demonstration adding the rare earth silicide wire results in a weld metal which contains over 0.50 percent silicon. These welds show significant reduction in notch toughness when compared to welds made without the additions. It is not known whether this loss in ductility can be attributed to silicon, to rare earths or is due to their combined influence. Therefore, the most desirable next welding tests should be with additions which do not contain elements that are known to influence mechanical properties significantly. This infers the need to proceed with the fabrication of wire which contains only misch metal.

FUTURE WORK

Plans for the immediate future of the program to evaluate the usefulness of rare earth additions for reduction of hydrogen embrittlement in HY-130 weld are threefold.

- (1) Produce cored electrode wire containing only rare earths by application of the fabrication knowledge contained in this report. (see page 18)
- (2) Examine wire adding techniques in an effort to aid the mixing of the special electrode with the basic filler metal and develop other improvements

in rare earth addition methods.

- (3) Utilize the wire produced and the technique developed to determine more precisely the effect of rare earths in HY-130 weld metal properties.