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DEVELOPMENT OF A WELDABLE HIGH STRENGTH STEEL (VIM/ESR PROCESSI--ETC(U)
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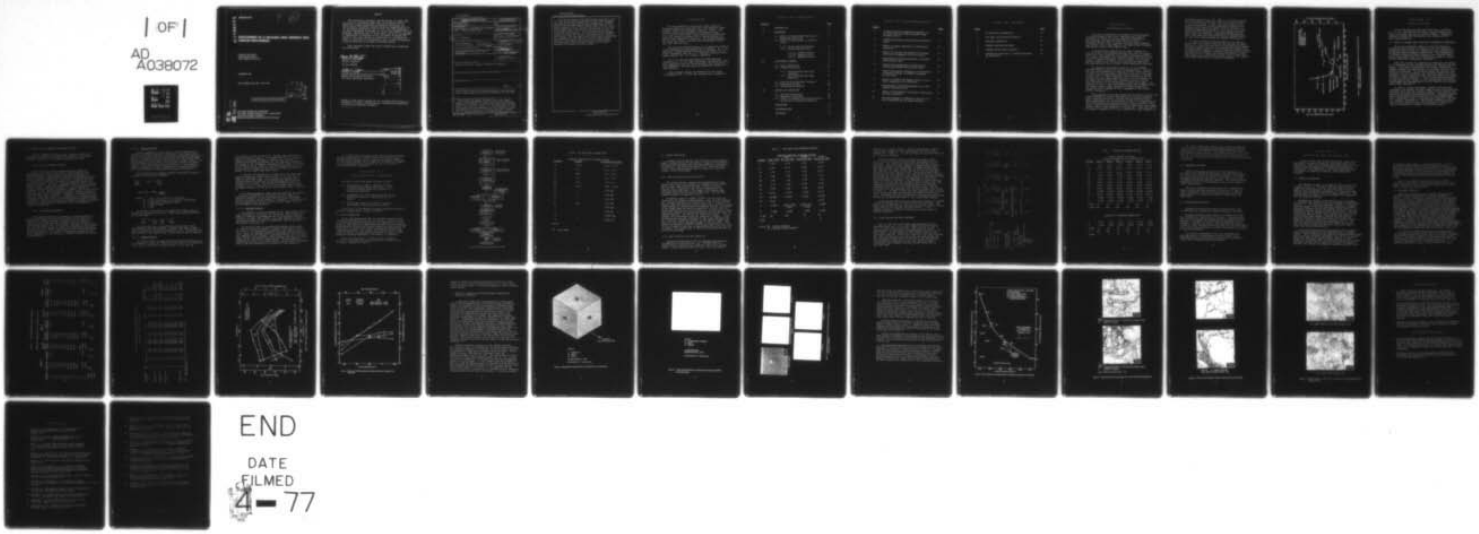

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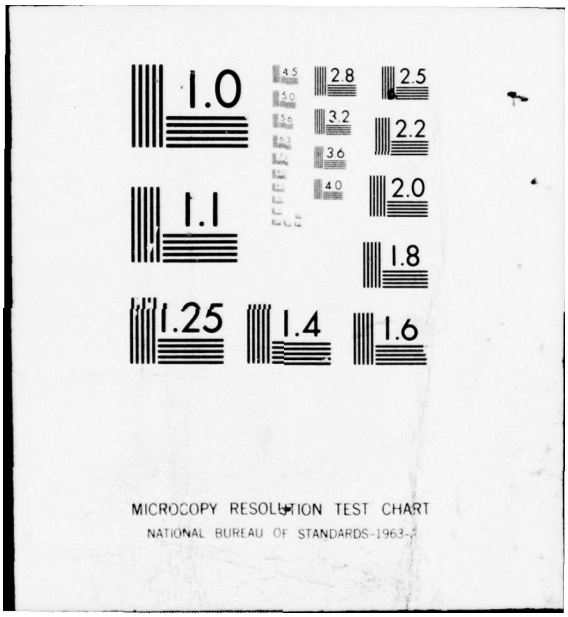
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DEVELOPMENT OF A WELDABLE HIGH STRENGTH STEEL (VIM/ESR PROCESSING)

GENERAL DYNAMICS
FORT WORTH DIVISION
FORT WORTH, TEXAS 76101

DECEMBER 1976

FINAL REPORT JULY 1975 - JULY 1976

Approved for public release; distribution unlimited

AIR FORCE MATERIALS LABORATORY
AIR FORCE WRIGHT AERONAUTICAL LABORATORIES
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This technical report has been reviewed and is approved for publication.

Martin H. Horowitz
Project Engineer

FOR THE COMMANDER

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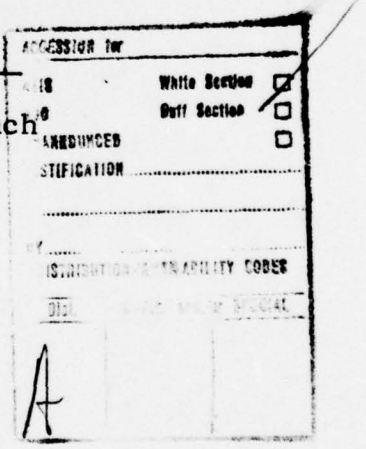

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A recently developed, VIM/VAR melted, high strength-toughness 14Co-10Ni-2Cr-1Mo-0.16C alloy steel (AF 1410) was selected as a candidate to evaluate the electroslag remelting process for high purity alloys. The principal goals in this investigation were the improvement in melt production economy and flexibility inherent to the ESR process with a minimal loss in toughness properties at desired strength levels.

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Six VIM electrodes of 14Co-10Ni-2Cr-1Mo-0.16C alloy steel (AF 1410) were electroslag remelted by single phase AC power with varying CaF_2-Al_2O_3-CaO-MgO flux compositions. VIM/ESR processed AF 1410 steel plate, aged at 950°F (810.0°C) for 5 hours, exceeded a TUS of 230 Ksi (1585.6 MPa); but as a result of the higher than normal inclusion content did not meet the CVN absorbed energy, ≥ 35 ft-lb (47.4J) required for a $K_{Ic} \geq$ 115 Ksi $\sqrt{in}$ (126.3 MPa $\sqrt{m}$). The resultant sulphur and oxygen content was higher than normally experienced in the VIM/VAR melted steel. The high oxygen level (76-350 ppm),in particular, was responsible for the excessive inclusion content which resulted in decreased energy required for ductile rupture. The non-metallic inclusions were primarily identified as manganese and chromium containing oxides.

F O R E W O R D

The work reported in this final report, which is designated Report No. AFML-TR-76-217, was performed under Contract F33615-73C-5093/P00006, Task No. 735105. This contract with General Dynamics' Fort Worth Division (P. O. Box 748, Fort Worth, Texas 76101) was accomplished under the technical direction of Mr. Martin Horowitz of the Air Force Materials Laboratory, LLS, Wright-Patterson Air Force Base, Ohio. All work was accomplished between July 1975 and July 1976.

The role and responsibilities of the personnel involved in the execution of this program are as follows: Dr. P. M. Machmeier, Program Manager-Principal Investigator; Mr. R. L. Jones, Mechanical Test; Mr. W. S. Margolis, Electron Microprobe Analysis; Mr. M. S. Howeth, reporting.

Dr. G. K. Bhat and associates at Nutek Materials Corporation (P. O. Box 4338, Pittsburgh, Pennsylvania 15204) were responsible for the VIM processing, flux preparation and composition, ESR processing and subsequent reduction to plate product.

This technical report was submitted by the author in September 1976. It has been reviewed and is approved.

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SECTION I

INTRODUCTION

A previous investigation concluded in the development of a high strength alloy steel combining high fracture toughness and stress corrosion properties, Reference 1. The resultant 14Co-10Ni-2Cr-1Mo-0.16C steel alloy, designated AF1410 steel, possesses high fracture toughness, $K_{Ic} > 130$ Ksi $\sqrt{in}$ (> 142.8 MPa $\sqrt{m}$), at high ultimate strength, 230-250 Ksi (1585.6-1723.5 MPa) levels.

At normal aging temperatures the high toughness of this alloy steel is partially derived from: (1) the precipitation of a fine dispersion of secondary hardening carbides in a highly dislocated lath martensite matrix and (2) the low levels of impurity elements. The low levels, of solid and gaseous elements were achieved by selection of high grade melting materials, and by double vacuum (VIM/VAR) processing.

The resultant mechanical properties of the VIM/VAR processed AF1410 steel alloy met all program goals. However, the cost and availability of high purity melt stock and cost of double vacuum processing could slow the general acceptance of this steel. In VAR processing, alloy segregation and the tendency for poor ingot surface increases as the diameter of the ingot and the molten pool increases, possibly setting some limitations on the processing of large scale ingots. In contrast to this, it has been demonstrated that the ESR process can produce smooth ingot surfaces and reduce nonmetallic inclusions with a minimum of alloy segregation and internal defects, Reference 2.

Investigations of the ESR processing of stainless steels and nickel superalloys reveal improvements in fracture toughness, short transverse ductility, and the fatigue endurance limit. These improvements can be attributed in part to a combination of the following factors: decreased sulfur content (sulfide inclusions), improved solidification structure (decreased alloy segregation), and a finer dispersion of inclusions. Since the levels of impurity elements (S, O, etc.) required for high toughness in

the AF1410 steel are low, Ref. Table 1, it was not expected that ESR processing could meet these levels, particularly in the case of oxygen. Rather, the improvements in microstructure obtained by ESR were expected to compensate for slightly higher levels of impurity elements resulting in a comparable toughness alloy steel. As a result, an investigation into the feasibility of electroslag remelting the AF1410 steel was initiated. Ultimately, EF-ESR processed steel, that would meet established material property levels for this steel, should provide the maximum cost benefits.

In this investigation ESR processing did not substantially reduce the sulfur content but did increase the oxygen content to a level where the effect of microstructural improvements were negated, resulting in somewhat lower toughness for the AF1410 steel, Figure 1. Since no apparent problems occurred in melting or in subsequent mechanical processing, it appears that ESR could be a viable melt process for this steel, provided oxygen can be controlled to lower levels by flux adjustments and/or improvement in operating parameters.

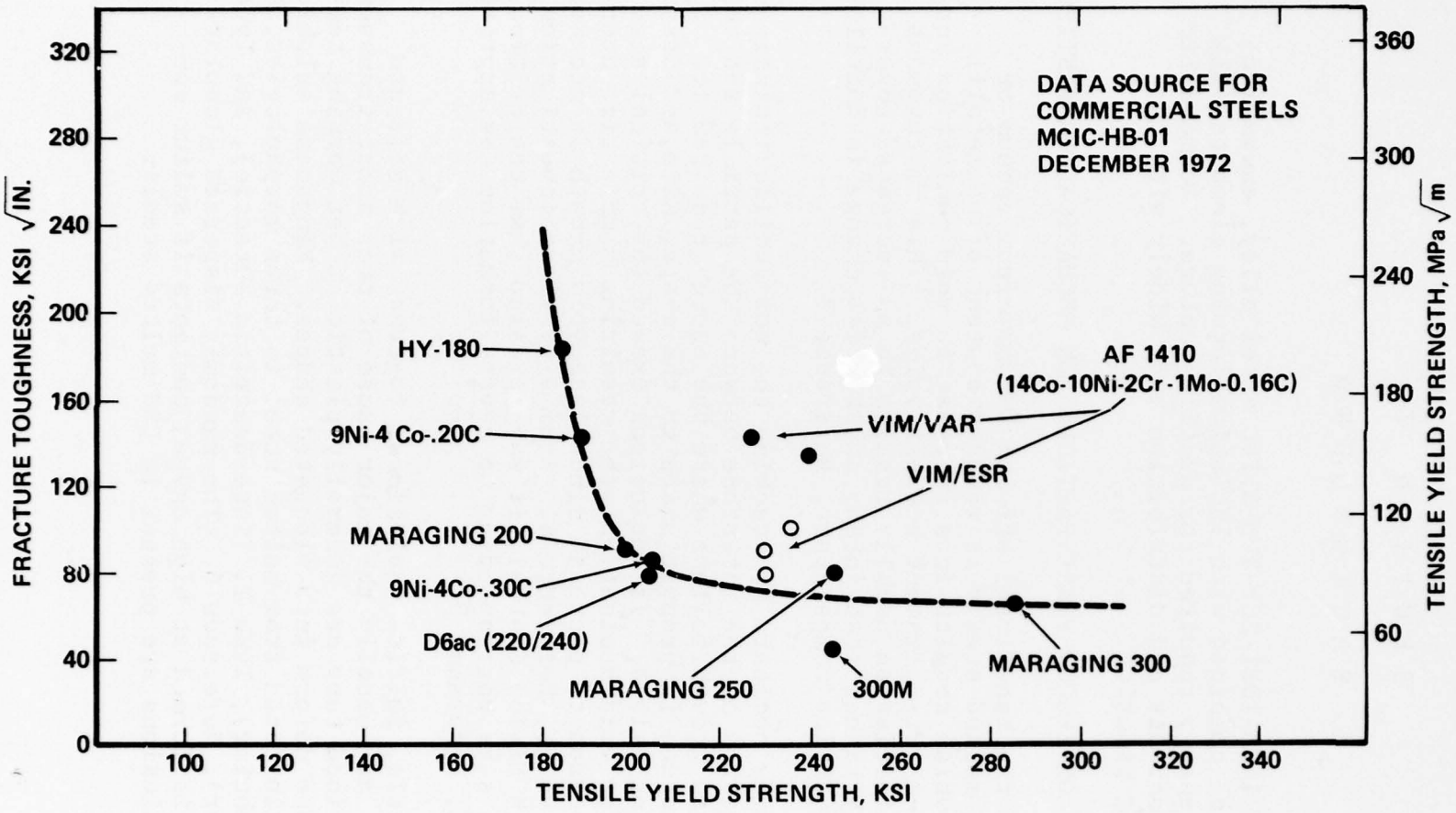


Figure 1 Strength – Fracture Toughness Comparison of AF 1410 Steel With Other Available High Strength Steels

SECTION II

BACKGROUND

In the 14Co-10Ni-2Cr-1Mo-0.16C steel alloy, excessive S, O, N, etc. combined with inclusion forming elements will reduce the energy required for ductile rupture. Nonmetallic inclusion contents and distribution vary widely with the steel making practice.

2.1 EFFECT OF SECOND PHASE PARTICLES ON MECHANICAL PROPERTIES

One of the beneficial effects derived from vacuum or electroslag melted steels is reduced content of nonmetallic inclusions which results in a decrease in void nucleation and an extended matrix ligament between voids. This improvement in fibrous initiation usually results in an increased upper-shelf energy rather than in any substantial change in ductile-brittle transition temperature, Reference 3.

Voids may nucleate by cracking the nonmetallic inclusions or by decohesion at the interface between the particle and the matrix. Void growth follows where the energy required for ligament rupture is proportionate to the shape, size,and distribution of inclusions. If inclusions exceed the critical size necessary for void nucleation, the resulting dimple size upon fracture is seen to decrease since less void growth is necessary to link up voids, Reference 4. From a previous investigation of aged high Ni-low C steel, it was revealed that the critical precipitate size corresponding to cavity formation was approximately 200Å, Reference 5.

In steels, sulfide inclusions, together with silicate inclusions, are usually the major cause of poor short transverse ductility since they are generally plastic at hot working temperatures and deform into elongated shapes. Manganese sulphide may be precipitated from molten steel in three morphologies, Type I (globular), Type II (interdentritic eutectic), and Type III (angular), Reference 6. The randomly dispersed globular Type I MnS is formed at high oxygen contents if sulfur containing inclusions are present in ESR melted steels.

2.2 EFFECT OF MELT PROCESS ON IMPURITY LEVELS

Impurity elements such as oxygen, nitrogen, sulfur, and hydrogen can usually be controlled or removed in the vacuum induction melting and electroslag remelting processes.

2.2.1 Vacuum Induction Melting

Vacuum induction melting enables precise control of temperature and pressure resulting in good control of the refining process. The control of the melting parameters in the VIM furnace are particularly effective in obtaining reduced levels of gaseous impurities in the steel. Elements which can be effectively removed or controlled by state-of-the-art VIM practices include: hydrogen - increased removal during boiling stage, nitrogen - high bath temperature - low presence of strong nitride forming elements, and oxygen - carbon deoxidation - formation of CO. Desulphurization is possible, but is not usually effective, during vacuum induction melting. Sulphur removal by slag additions (lime + fluospar) is possible at oxygen levels of less than 20-30 ppm, Reference 7. However, if the oxygen content increases above that level, the desulfurization reaction becomes reversible. Practices involving the removal of sulphur by forming volatile and solid sulfides generally lead to degradation of mechanical properties.

2.2.2 Electroslag Remelting

Electroslag remelting is a secondary refining process in which an electrode of the desired composition is melted in a electrically conductive slag. Droplets of molten metal are refined as they pass through the molten flux and solidify in a water-cooled copper mold. The primary advantages of this process are the efficient removal of sulfur and improved solidification structure in steel alloys, both which are not readily achievable in the VIM process. The effect of ESR process parameters and flux composition on the refining of impurity elements is briefly discussed below.

2.2.2.1 Sulphur Control

Sulfide capacities of CaF_2 mixtures with an optimum mole fraction of CaO will maintain a high level of desulphurization under highly reducing as well as oxidizing conditions, Reference 8. However, comparative melts conducted with CaF_2 and $70CaF_2$-30CaO demonstrated that both slags were inferior to ternary slags in the CaF_2-CaO-Al_2O_3 system based on ingot surface quality and lower globular oxide inclusion content, Reference 2. The transfer of sulphur from metal to slag is enhanced by a high slag basicity and a low atmospheric oxygen partial pressure, Reference 9.

The kinetics of net sulphur transfer from metal to slag is a first order reaction as follows:

$$\underset{\text{metal}}{|S|} + 2\bar{e} = \underset{\text{slag}}{(S^{-2})}$$

$$-\log \left|\frac{S}{S}\right| = 100A \frac{Km}{2.3\ Wm} t$$

where S = conc. of S initial/S at time (t)
K_m = rate coefficient of S transfer, metal/slag
A = slag/metal interfacial area
W_m = mass of metal
t = time

In contrast, the transfer of sulphur from slag to gas is favored by low slag basicity and a high oxygen partial pressure in the gas phase.

$$\underset{\text{slag}}{S^{-2}} + 3/2\ \underset{\text{gas}}{O_2} = \underset{\text{gas}}{SO_2} + \underset{\text{slag}}{O^{-2}}$$

The fact that sulfur removal does not take place under argon suggests that the transfer of oxygen from slag to metal aids the transfer of sulphur from metal to slag. The AC-ESR process results in less ingot sulfur content than the DC-ESR process.

2.2.2.2 Oxygen Control

The main sources of oxygen during electroslag remelting are (1) atmospheric oxygen (2) electrode oxide scale (3) environmental water vapor or chemically bonded moisture in the flux and

(4) reducible oxides in the flux. Melting under atmospheric conditions causes oxygen to be absorbed directly by the liquid slag at the gas/slag boundary or passes into the slag via reaction with sulfur, Reference 10. The slag then transports the oxygen to the slag/metal interface where oxygen is absorbed by the liquid metal. However, the final oxygen content in the molten steel may depend on the level of deoxidizing elements present in the liquid slag and/or steel.

To prevent the absorption of oxygen into the liquid metal strong deoxidizing elements such as Si, Ti, Al, etc. can be added to the molten slag. This procedure is particularly effective when the oxides of the deoxidizing specie are not contained in the slag. ESR melting of stainless steels containing 0.5 to 0.7% (Al, Si, Ti) in the liquid metal resulted in oxygen contents of 9-34 ppm in the resultant ingot, Reference 10.

Since high atmospheric oxygen partial pressures are related to increased absorption of the oxygen into the slag, it is sometimes expedient to melt under protective gas shielding. However, decreased sulfur removal is a penalty. AC operation and DC superimposed over AC will result in lower oxygen contents than straight DC-ESR operation.

2.2.2.3 Hydrogen Control

The tendency to pick up hydrogen in the electroslag remelted ingot increases as the ingot size increases. The sources and causes of hydrogen contamination are (1) atmospheric humidity (exposure of large slag surface), (2) slag composition (water in the slag materials and water of hydration), and (3) polarity of ESR process, Reference 11.

AC remelting leads to considerable hydrogen pickup from the calcium fluoride + alumina + calcium oxide slag to levels in excess of the equilibrium solubility at the melting point of iron. Thus, the hydrogen pickup is controlled by the reaction at the slag/metal pool interface. Remelting through CaF_2 or C_aF_2 + Al_2O_3 results in considerably lower hydrogen contents since these slags are not particularly deliquescent. Another approach to minimizing hydrogen is to remelt under a dry protective atmosphere with a slag also premelted or calcined in a dry atmosphere.

It is known that DC melting, while reducing hydrogen pickup, produces ingots with less sulphur removal and greater oxygen pickup than AC melting. Thus, it appears the optimum conditions for maintaining hydrogen at low levels and retaining the capacity for removing sulphur and oxygen will be the use of good AC remelting practice, Reference 12.

SECTION III

EXPERIMENTAL PROGRAM

The scope of this experimental program includes:

(1) The melting of a 2000 lb (909.1Kg) VIM heat of AF1410 and subsequent reduction to 7 inch (17.8cm) diameter electrodes suitable for electroslag remelting.

(2) Determination of flux compositions and melting parameters suitable for ESR melting of six ingots.

(3) The melting, conversion, thermal treatment, and testing of ESR laboratory size heats.

A flow chart of the VIM/ESR processing, mechanical reduction, and heat treatment steps is given in Figure 2.

3.1 ALLOY COMPOSITION

It has been demonstrated that the inherent toughness of the 14Co-10Ni-2Cr-1Mo-0.16C steel alloy is related to the low levels of residual and impurity elements that are obtainable by a combination of high purity melt ingredients and double vacuum processing. Maintaining the alloying and residual/impurity elements within the limits given in Table 1 will result in acceptable strength and toughness properties. Any sizeable deviation from these element levels during melt processing can be expected to result in some mechanical property degradation, particularly in toughness.

After ESR processing of six ingots, three candidates for further processing were selected based on the lowest level of impurity elements per Table 1.

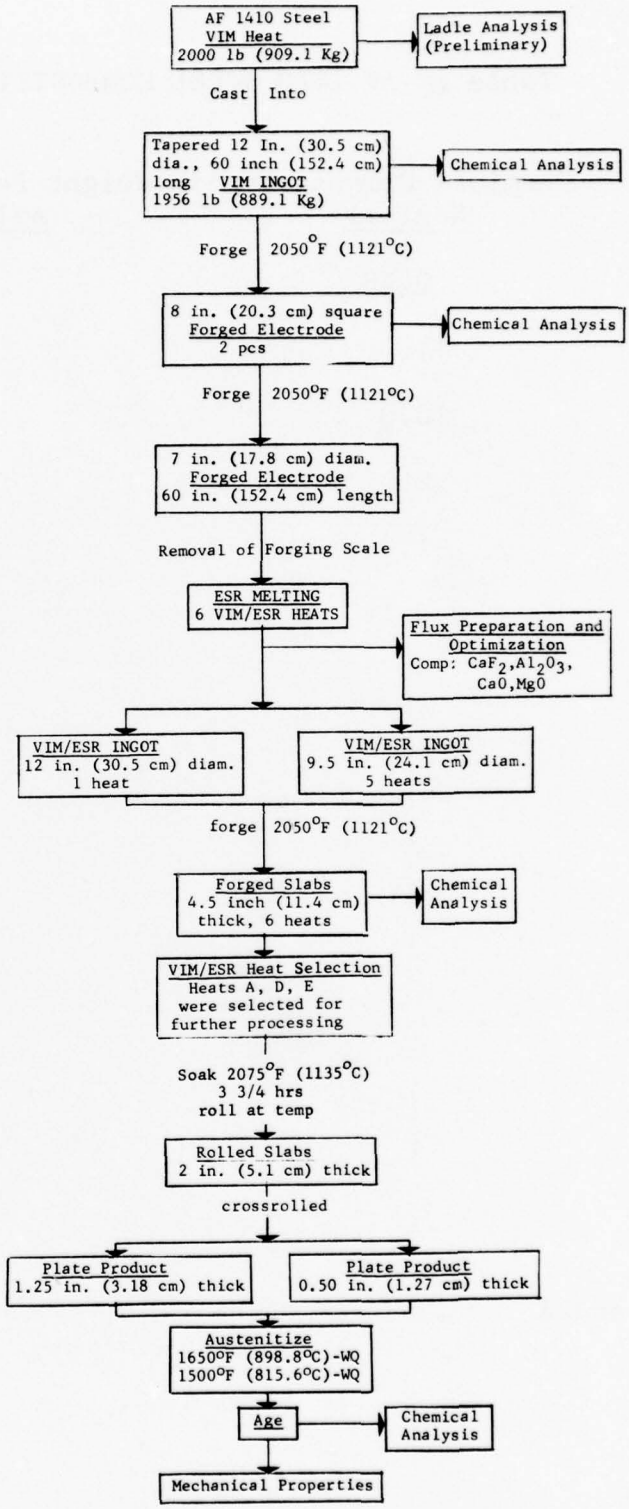


Figure 2 VIM/ESR Processing and Mechanical Reduction Steps

Table 1 AF 1410 STEEL COMPOSITION

Elements	Chemical Composition in Weight Percent Nominal	Allowable Variation
C	0.16	0.15 - 0.17
Co	14.0	13.5 - 14.5
Ni	10.0	9.5 - 10.5
Cr	2.0	1.8 - 2.2
Mo	1.0	0.9 - 1.1
Mn	0.15	0.05 - 0.20
Si	NA	0.10 max
Al		0.01 max
Ti		0.01 max
Zr		0.01 max
S		0.005 max
P		0.01 max
N		0.0025 max
O		0.003 max

bal Fe

NA - none added

3.2 VIM/ESR PROCESSING

A single VIM heat was cast into ingot form and subsequently forged to shape for the ESR electrodes. Ranges of flux compositions and melt parameters were identified which were expected to be conducive to the ESR processing of the AF 1410 steel composition. After ESR processing selected ingots were reduced to 2 inch (5.1 cm) thick slabs.

3.2.1 VIM Processing and Ingot Reduction

The vacuum induction melting was accomplished with high purity (low S and P) melt charges in a magnesia lined crucible. After a check ladle analysis the 2000 lb (909.1 Kg) melt was cast into a tapered 12 inch (30.5 cm) diameter 60 inch (152.4 cm) long ingot mold. The weight of the tapered ingot including the hot top was 1956 lbs (889.1 Kg). The VIM ingot chemical analysis is reported in Table 2..

Following a reheat at 2050°F (1121°C), the VIM ingot was forged into two lengths of approximately 7 inch (17.8 cm) diameter bar. During forging the hot top portion of the 12 inch (30.5 cm) diameter ingot was removed and later forged into a 1 inch (2.54 cm) thick pancake for starter material during ESR melting. The balance of the forged ingot was further reduced into a 8 inch (20.3 cm) square bar which was cut into two sections. At this stage of forging several samples of material were removed from a shear slice for chemical analysis. The results are reported in Table 2. All the analyses were reproducible with the exception of oxygen. It was speculated that the wide variations in the oxygen analysis could be a function of sampling technique so that greater credence was given the less than 25 ppm value evident in the VIM ingot. Each section was reheated at 2050°F (1121°C) and forged into two 7 inch (17.8 cm) diameter round bars of approximately 60 inches (152.4 cm) in length. The forging scale on each bar (electrode) was removed prior to ESR melting.

3.2.2 ESR Processing and Ingot Reduction

Electroslag remelting of the six designated ESR heats was accomplished at Nutek Materials, Inc. The ESR facility is powered with an industrial size 1000 KVA (30-70 volt) AC power source. Single phase AC power was used in this investigation

Table 2 VIM INGOT AND ELECTRODE ANALYSIS

	Chemical Analyses in Weight Percent			
Element	Ingot (CM)	Ingot Hot Top (CM)	Forged Electrode (CM)	Forged Electrode (NS)
C	0.155	0.16	0.16	0.18
Co	13.61	14.14	14.09	14.24
Ni	9.65	9.73	9.94	10.74
Cr	1.98	1.96	1.93	1.91
Mo	1.08	1.02	1.08	1.10
Mn	0.14	0.06	< 0.01	0.16
Si	0.09	0.04	0.02	< 0.01
Al	< 0.01	0.016	0.016	< 0.01
Ti	< 0.008	< 0.005	< 0.005	< 0.005
Zr	< 0.01	-	-	< 0.01
S	0.005	0.006, 0.008 0.010	0.007, 0.007 0.008	0.008
P	0.005	0.006	0.006	0.003
O ppm	20	250	26	112
N ppm	7	-	-	8

Notes: CM - Cannon Muskegon
NS - National Spectrographic

primarily for oxygen control. During remelting the range of current and voltage was 4650-5900 amperes and 37-40 volts, respectively, Table 3. In all the melts a cold start was used as a start-up technique.

Using low sulfur practice attempts were made to ESR the AF 1410 steel alloy with high CaF_2 containing flux compositions. The first flux composition (92 CaF_2-bal Al_2O_3) developed a high resistance causing a rippled surface on the ESR ingot and had to be terminated. A 88CaF_2-10Al_2O_3-2MgO flux improved the ingot surface quality but did not increase the melt rate to the desired level. These experiments indicated that the CaF_2 content of the slag should not be increased beyond 85% and also that the Al_2O_3 content be increased from 10%. Since difficulty resulted in the separation of the joined electrode ingot by cutting, it was decided to remelt the first ingot in a 12 inch (30.5 cm) diameter water cooled copper mold. Subsequent remelting of the remaining five electrodes was accomplished in 9.5 inch (24.1 cm) diameter molds. The six electrodes were ESR processed with the flux compositions listed in Table 3. Several of the heats were remelted using an argon cover, especially when melting was occurring in the hot top area. No attempt was made to maintain a deoxidizing slag during melting but aluminum was added to the slag during charging.

Following ESR processing the ingots were reheated at 2050°F (112°C) and forged into 4.5 inch (11.4 cm) thick slabs. The slabs were hot sheared into two sections with the center section being retained for chemical analysis, Table 4. The chemical analysis resulted in selection of three heats for further reduction.

3.3 PLATE ROLLING AND HEAT TREATMENT

Two 4.5 inch (11.4 cm) slabs representative of the selected heats (A, D, and E) were heated at 2075°F (1135°C) for 3 3/4 hours prior to rolling. Each slab was rolled straight-away in nine passes to approximately 2 inch (5.1 cm) thick plate. At this stage the plate product was cut into four equal sections. Two of the 2 inch (5.1 cm) thick sections were cross-rolled into 1.25 inch (3.18 cm) thick plates and the remaining sections into 0.5 inch (1.27 cm) thick plates. The rolling was conducted in such a way that both plate thicknesses were produced from the top and bottom half of each ESR ingot.

Table 3 ESR MELT CONDITIONS

Parameters	Heats A	B	C	D	E	F
Forged electrode dia., in.	7	7	7	7	7	7
(cm)	(17.8)	(17.8)	(17.8)	(17.8)	(17.8)	(17.8)
Mold diameter, in.	12	9.5	9.5	9.5	9.5	9.5
(cm)	(30.5)	(24.1)	(24.1)	(24.1)	(24.1)	(24.1)
Ingot weight, lbs.	317	334	218	278	276	298
(Kg)	(144.0)	(151.8)	(99.1)	(126.4)	(125.5)	(135.5)
Melting Parameters						
Current, amperes	5700	5900	4900	4650	4700	4500
Voltage, volts	40	38-39	36-37	38-39	37-38	40
Melting time, min.	40	35	23	35	37	35
Flux Preparation and Composition						
Flux weight, lbs.	58	42	42	47	47	47
(Kg)	(26.4)	(19.1)	(19.1)	(21.4)	(21.4)	(21.4)
Molten flux preparation time, min.	8	10	11	13	13	11
Flux drying	1500°F (815.6°C) for 8 hours minimum					
Flux Composition, percent						
CaF_2	80	72	70	70	70	75
Al_2O_3	15	24	26	22	20	15
CaO	3	4	-	6	8	7
MgO	2	-	4	2	2	3

Table 4 VIM/ESR ELECTRODE ANALYSES

Forged VIM/ESR Electrodes
Chemical Composition in Weight Percent

Element	Heat A	Heat B	Heat C	Heat D	Heat E	Heat F
C	0.13	0.16	0.17	0.20	0.19	0.19
Co	13.35	14.02	14.09	14.07	14.11	14.21
Ni	9.73	10.53	10.68	10.42	10.66	10.79
Cr	1.70	1.82	1.88	1.87	1.88	1.87
Mo	1.06	1.15	1.17	1.10	1.13	1.13
Mn	0.14	0.13	0.15	0.16	0.16	0.14
Si	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Al	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Ti	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
Zr	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
S	0.005	0.007	0.008	0.008	0.007	0.007
P	0.003	0.004	0.002	0.003	0.003	0.003
N ppm	12	12	11	5	11	11
0 ppm	184	226	100	148	181	177

RECHECK OF ELECTRODE COMPOSITION

C	0.15	0.15	0.14	0.17	0.16	0.14
S	0.005	0.007	0.007	0.006	0.006	0.007
N ppm	8	10	10	10	8	8
0	350	220	76	100	87	170
H ppm	1	1	<1	1	<1	1

The cross-rolled plate product was double austenitized prior to aging. The double austenitizing sequence consisted of heating to 1650°F (898.8°C) and 1500°F (815.6°C) with a water quench at each interim. The soaking time was 1 1/4 hours for the 1.25 inch (3.18 cm) thick plate and approximately 3/4 hour for the 0.5 inch (1.27 cm) thick plate at each austenitizing temperature.

3.4 MECHANICAL TESTING

Tensile tests were conducted at room temperature in accordance with ASTM E8-68 and Federal Test Method Standard No. 151a using a 0.252 inch diameter specimen. Tests were performed in both the L-T and T-L plate orientation using a 120,000-pound-capacity BLH test machine. The yield point was determined with a PS-5M extensometer with a strain rate of 0.003 inch/inch/min.

The notch toughness was determined by a standard-size Charpy V-Notch specimen sectioned in both the L-T and T-L plate orientation. The testing was conducted in a Riehle Impact machine at ambient and cryogenic temperatures per ASTM 370A-1.

3.5 MFTALLURGICAL ANALYSIS

Standard polishing and etching techniques were used in preparing the specimen for optical microscopy analyses.

Unetched specimens were prepared by diamond polishing for examination of nonmetallic inclusions. Selected areas were further analyzed by electron microprobe (ARL Model AMX) analysis. The samples were examined for manganese, iron, chromium, aluminum, and oxygen. The analysis was primarily limited to x-ray intensity profiles across the inclusion field. Point counting was required to confirm the presence of oxygen in various inclusions.

The fracture topography of the test specimens were analyzed both at the macroscopic and microscopic level. High magnification fractographic analysis was performed on a JEOL JSM-2 Scanning Electron Microscope.

SECTION IV

RESULTS AND DISCUSSION

A 2,000 lb (909.1 Kg) VIM heat was melted, forged to six electrodes, and remelted using six flux compositions by the ESR process. Three of the VIM/ESR ingots were selected on the basis of chemical composition for additional processing to rolled plate. The properties of the cross-rolled plate were investigated by chemical, mechanical, and metallurgical analysis.

4.1 CHEMICAL COMPOSITION

The chemical analysis of the 2000 lb (909.1 Kg) VIM heat is given in Table 2. The major alloying elements met the composition requirements for AF 1410 steel given in Table 1. However, based on numerous evaluations, the sulfur and oxygen contents were 0.005-0.01% and 20-250 ppm, respectively. The wide variation in oxygen content is thought to be associated with the sampling technique.

Following the forging of the VIM ingot into electrodes, six ESR heats were melted with the flux compositions designated in Table 3. The chemical composition in Table 4 indicates that few changes in the major alloying elements were evident. Apparently chromium was the only alloying element subject to a consistent loss when passing through the slag layer. With the exception of Heat A, sulfur was not reduced from the starting value during ESR processing. This behavior was judged to be unusual since slags containing greater than 50% CaF_2 are generally reported to remove up to 60% of the sulfur content. Oxygen was picked up during ESR processing, Table 4. The levels of oxygen encountered were much higher (76-350 ppm) than expected since selected heats were ESR melted under an argon cover with AC-ESR operation.

As was previously discussed, deoxidizers must be added to either the metal composition or slag composition for oxygen control. The AF 1410 alloy steel which is vacuum-carbon-deoxidized does not contain sufficient deoxidizers (Al, Ti, Si) to prevent oxygen from entering the molten metal. Addition of these elements to the molten steel, in sufficient quantities to control the oxygen content, may not result in the level of toughness desired. As a result, the alternative approach would be to add

strong deoxidizing elements to the slag composition. In this investigation Al was added to the flux composition at melt start-up but additions were not made during the melting operation. Electroslag remelting of 10Ni-Cr-Mo-Co type steels, using periodic deoxidizing additions to the molten slag, has demonstrated that oxygen levels as low as 32-45 ppm can be achieved, Reference 13. In all of the VIM/ESR heats hydrogen was maintained at very low levels.

Three of the VIM/ESR heats were selected for further reduction to rolled plate. The chemical analyses of the rolled plate confirmed the loss of chromium and the previously reported levels of oxygen and sulfur, Table 5.

4.2 MECHANICAL PROPERTIES

Rolled plate tensile and notch toughness properties were obtained for three VIM/ESR heats in five heat treatments, Table 6. It was not possible to compare the VIM ingot properties with previous melts as Nutek Materials Corporation had difficulty in obtaining a section suitable for test material. Data representative of specimens aged at 950°F (510.0°C) revealed that the notch toughness of heats A, D, and E did not meet the required absorbed energy 35 ft-lbs (47.4J) required for a $K_{Ic} \geq 115$ Ksi $\sqrt{in}$ (126.3MPa $\sqrt{m}$). However, the VIM/ESR rolled plate specimens exceeded the strength requirement of 230 Ksi (1585.6MPa) TUS while exhibiting a minimum of anisotropy. When the properties of AF 1410 VIM/ESR rolled plate (Heat E) are compared to VIM/VAR processed material, it is evident that both the ultimate tensile strength and notch toughness are reduced over a wide range of aging temperatures, Figure 3. In contrast, the yield strength is increased from the VIM/ESR processed steel over the entire range of aging temperatures. This behavior results in decreased strain hardening at 950°F (810.0°C) aging temperature as the TYS/TUS ratio increased from 0.93 to 0.97 for the VIM/VAR and VIM/ESR processed AF 1410 steel, respectively.

The effect of VIM/ESR processing had a pronounced effect on the upper shelf absorbed energy while little effect was noted in the energy required for ductile tearing at cryogenic temperatures, Figure 4. An excessive content of sulfur and oxygen, albeit a fine dispersion, was responsible for the decrease in upper shelf energy of approximately 35%. This behavior has been previously described in steels in which progressively higher

Table 5 VIM/ESR ROLLED PLATE ANALYSES

Chemical Composition in Weight Percent

	Heat A			Heat D				Heat E	
Element	Electrode	Recheck	VIM/ESR Rolled Plate	VIM/ESR Electrode	Electrode Recheck	VIM/ESR Rolled Plate	VIM/ESR Electrode	Electrode Recheck	VIM/ESR Rolled Plate
C	0.13	0.15	0.15	0.20	0.17	0.15	0.19	0.16	0.16
Co	13.35		13.40	14.07		13.67	14.11		13.76
Ni	9.73		9.23	10.42		9.66	10.66		9.64
Cr	1.70		1.46	1.87		1.79	1.88		1.72
Mo	1.06		0.91	1.10		1.12	1.13		1.07
Mn	0.14		0.14	0.16		0.18	0.16		0.17
Si	<0.01		<0.01	<0.01		<0.01	<0.01		<0.01
Al	<0.01		<0.01	<0.01		<0.01	<0.01		<0.01
Ti	<0.005		<0.01	<0.005		<0.01	<0.005		<0.01
Zr	<0.01		-	<0.01		-	<0.01		-
V	-		<0.01	-		<0.01	-		<0.01
S	0.005	0.005	0.003	0.008	0.006	0.006	0.007	0.006	0.007
P	0.003		0.010	0.003		0.010	0.003		0.014
N ppm	12	8	13	5	10	11	11	8	<5
O ppm	184	350	320	148	100	220	181	87	88
H ppm	-	1	2	-	1	2	-	<1	4

Table 6 MECHANICAL PROPERTIES OF VIM/ESR PROCESSED AF 1410 STEEL

Cross-Rolled 0.50 Inch (1.27 cm) Thick Plate

Heat Treatment Condition	Heat No.	Orientation	TYS Ksi	TYS (MPa)	TUS Ksi	TUS (MPa)	% Elong. 4D	% RA	Charpy V-Notch Absorbed Energy ft-lbs	Charpy V-Notch Absorbed Energy (Joules)
A/Q	A	LT	205.9	(1419.6)	237.5	(1637.5)	12	54.6	27.2	(36.9)
	D	LT	196.0	(1351.4)	231.8	(1598.2)	12	57.1	27.1	(36.7)
	E	LT	218.1	(1503.7)	241.9	(1667.8)	13	58.5	32.9	(44.6)
850°F (454.4°C)/ 5 hr	A	LT	229.1	(1579.6)	242.3	(1670.6)	13	52.8	21.5, 21.7	(29.2)(29.4)
	D	LT	230.6	(1589.9)	248.5	(1713.3)	13	52.1	23.0, 23.4	(31.2)(31.7)
	E	LT	237.5	(1637.5)	253.3	(1746.4)	12	55.2	26.1, 26.3	(35.4)(35.7)
900°F (482.2°C)/ 5 hr	A	LT	239.2	(1649.2)	248.1	(1710.6)	12	55.5	22.7, 23.4	(30.8)(31.7)
	D	LT	245.5	(1692.7)	254.5	(1754.7)	13	57.1	25.8, 27.4	(35.0)(37.1)
	E	LT	247.9	(1709.2)	255.9	(1764.4)	12	57.5	28.0, 28.9	(38.0)(39.2)
950°F (510.0°C)/ 5 hr		LT	231.4	(1596.4)	235.4	(1623.0)	12	57.5	25.2	(34.2)
	A	LT	227.6	(1569.2)	234.6	(1617.5)	12	58.5	23.8	(32.3)
	A	TL	237.0	(1634.1)	241.0	(1661.6)	12	52.7	24.2, 25.0	(32.8)(33.9)
	D	LT	231.1	(1593.4)	235.4	(1623.0)	13	50.5	28.7	(38.9)
	D	LT	229.2	(1580.3)	234.1	(1614.1)	12	58.8	29.2	(39.6)
	D	TL	235.0	(1620.3)	237.0	(1634.1)	13	54.9	27.3, 28.3	(37.0)(38.2)
	E	LT	235.0	(1620.3)	241.0	(1661.6)	12	60.0	31.9	(43.3)
	E	LT	231.8	(1598.2)	235.8	(1625.8)	13	57.1	28.6	(38.8)
	E	TL	232.7	(1604.4)	236.7	(1632.0)	12	60.4	28.2, 28.8	(38.2)(39.0)
1000°F (537.8°C)/ 5 hr	A	LT	207.5	(1430.7)	211.4	(1457.6)	12	60.8	25.0, 25.8	(33.9)(35.0)
	D	LT	205.2	(1414.8)	207.2	(1428.6)	12	61.1	28.1, 28.4	(38.1)(38.5)
	E	LT	205.3	(1415.5)	206.3	(1422.4)	12	57.8	25.8, 28.0	(35.0)(38.0)

Notes: Double austenitized at 1650°F (898.8°C)-WQ/1500°F (815.6°C)-WQ prior to aging
Room temperature data

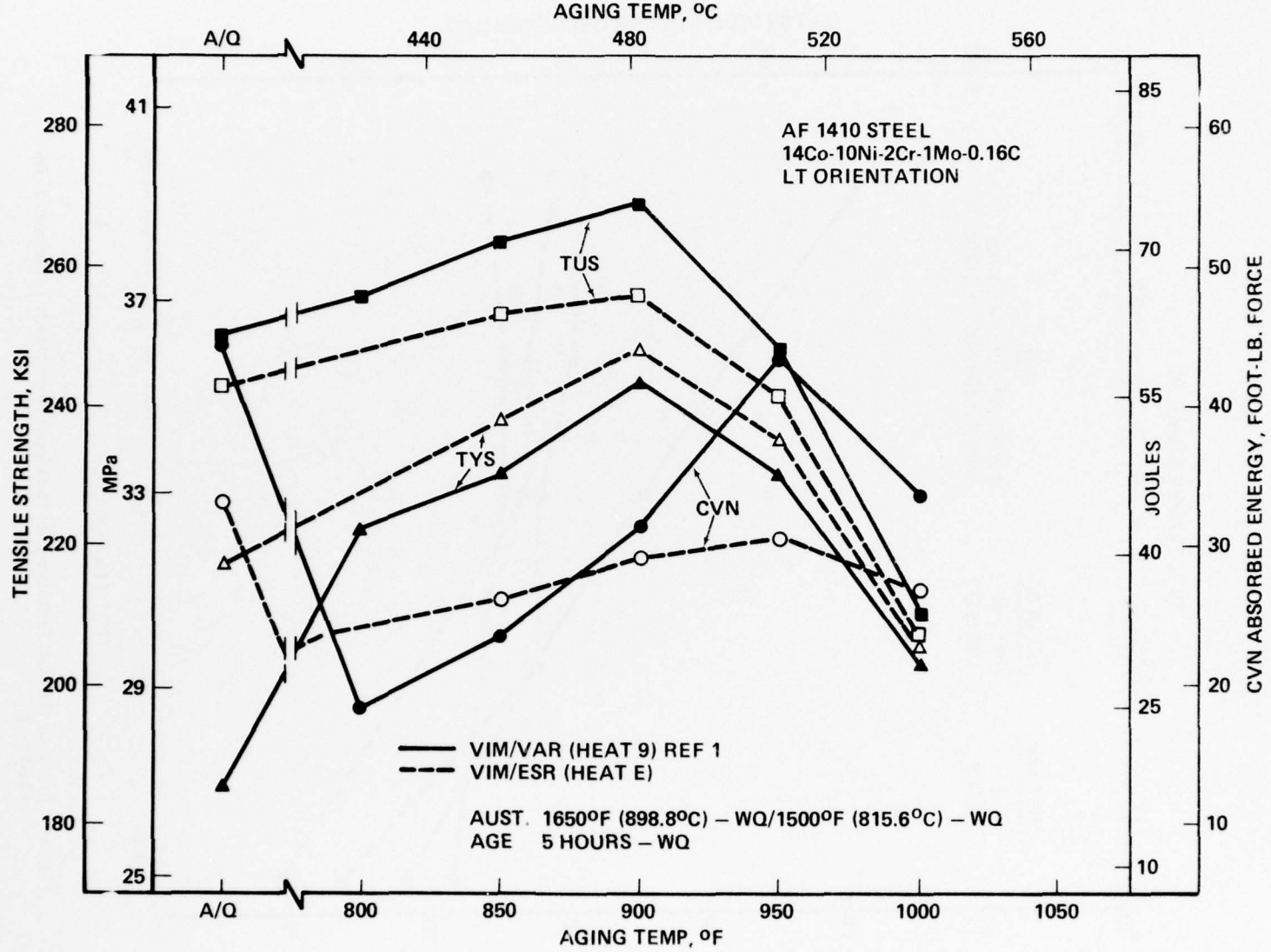


Figure 3 Effect of Aging Temperature on Mechanical Properties

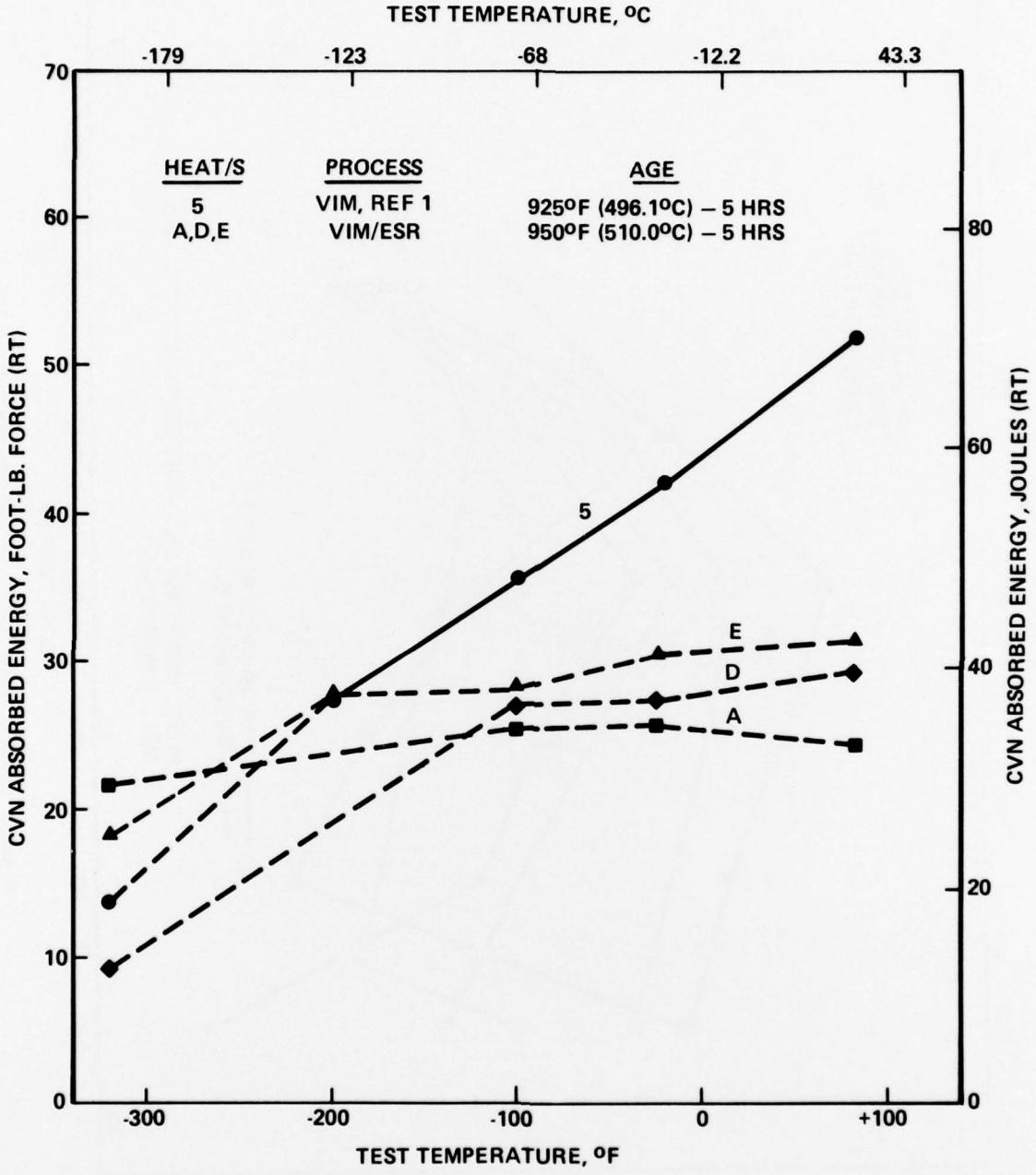


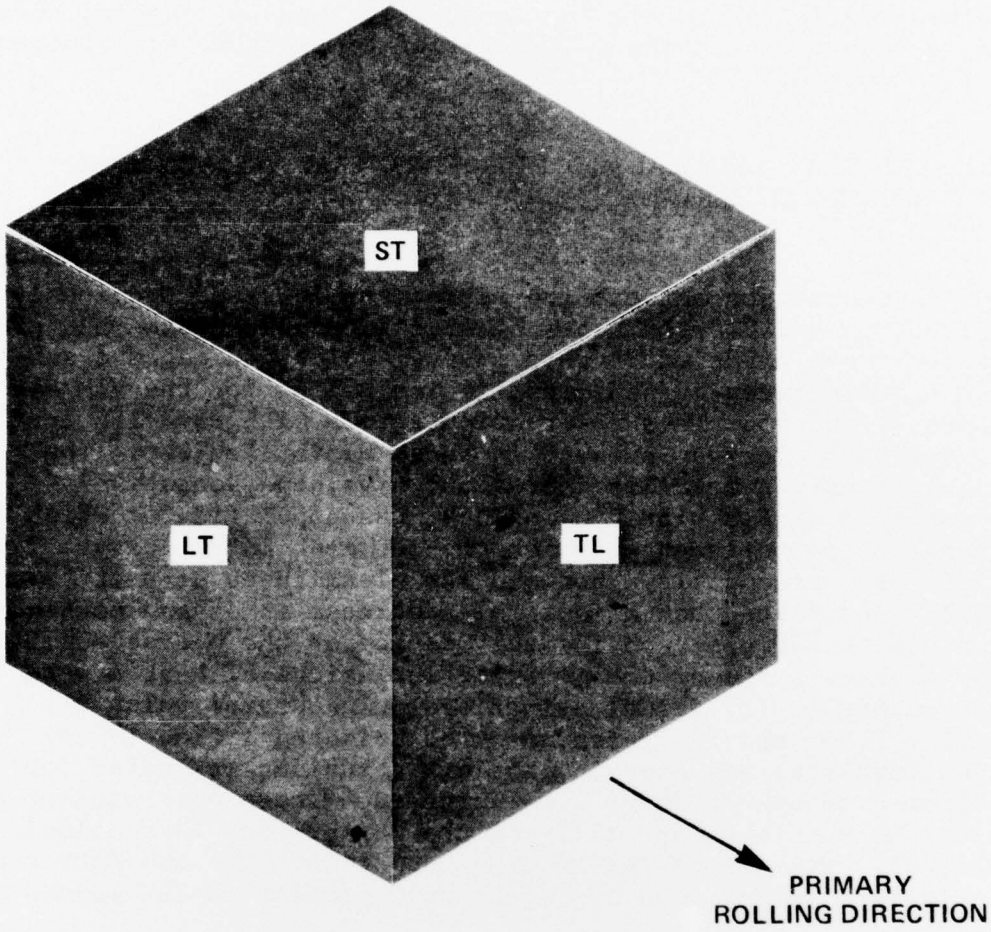
Figure 4 Effect of the Melt Processing On the Upper Shelf Notch Toughness of AF 1410 Steel

values of sulfur has produced progressively decreased energy values for fully ductile ruptures, Reference 14. Again, there was a moderate decrease in transition temperature with increasing sulfur content.

4.3 EFFECT OF CHEMICAL AND MICROSTRUCTURAL VARIATIONS ON MECHANICAL BEHAVIOR

A comparison between the microcleanliness of previously melted VIM and VIM/VAR heats of AF 1410 steel and the ESR processed steel produced in this investigation indicated a substantial increase in non-metallic inclusions for the VIM/ESR heats, Figure 5. This observation agrees with the high level of sulfur and oxygen present in these heats, Table 5. The majority of non-metallic inclusions have been tentatively identified as globular type oxides, the population of which increases proportionately with the increase in the oxygen level, Figure 6. A comparison between the cleanliness of heat E and heat A indicates that a four fold increase in oxygen content (88 ppm to 350 ppm) considerably decreases the microcleanliness. The average sizes of the globular inclusions of heat A were determined by (1) visual comparison-Jernkontaret value was D3 at 100X ($<7.2\mu$) and (2) measurement of selected fields ($5\text{-}8\mu$). A uniform size and distribution of inclusions was noted for the polished specimens which were surveyed. These observations agree with the reported capability of the ESR process for inclusion control. With minor variances due to ingot size and melt rate, inclusions larger than 10μ are rarely present in ESR melted steels, Reference 15.

Electron microprobe analysis of the VIM/ESR processed AF 1410 steel (Heat A) disclosed that the non-metallic inclusions were primarily manganese and chromium containing oxides, Figure 7. The presence of oxygen was confirmed by point counting. For the fields selected, the presence of aluminum oxides and manganese sulfides was not verified. With relatively low quantities of the strong oxide forming elements (Al, Ti, etc.) in the AF 1410 steel composition it follows that chromium and manganese should tie up the oxygen. In fact chromium may have played the role of a weak deoxidizing element in the molten metal during the ESR melting as the ingot chemistry of all the heats indicate some loss of this element to the slag. It is suspected that the sulfur was present in globular Type I sulfides since no elongated inclusions were noticed after hot rolling. It has been reported



HEAT E

S – 0.007 WT. %
N – 8 ppm
O – 88 ppm

MAGNIFICATION: 100X

JK RATING D2 – THIN SERIES

Figure 5 Representative Microcleanliness of VIM/ESR Cross-Rolled Plate

HEAT A
S – 0.005 WEIGHT PERCENT
N – 8 ppm
O – 350 ppm

TL ORIENTATION
MAGNIFICATION: 100X

JK RATING D4 – THIN SERIES

Figure 6 Typical Microcleanliness of High Oxygen Containing VIM/ESR Cross-Rolled Plate

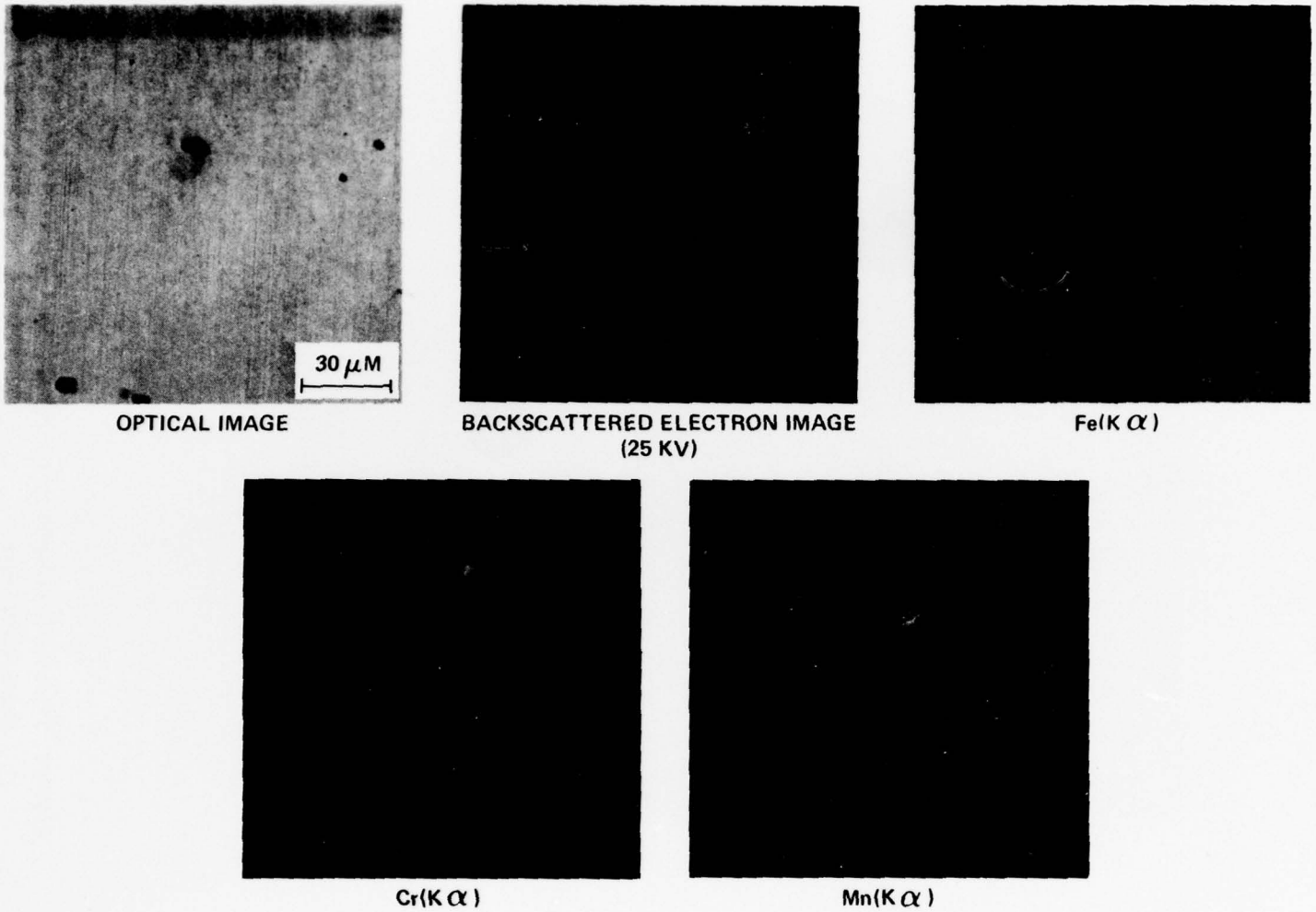


Figure 7 Electron Microprobe Analysis of Non-Metallic Inclusions Present in VIM/ESR Processed AF 1410 Steel

that the amount of oxygen in the solution has a marked effect upon the type of sulfides in the steel after solidification, Reference 16. As a result Type I sulfides form as isolated globular particles possessing the highest oxygen of the sulfide groups.

Excessive quantities of impurity elements (S, O) resulted in a substantial loss of notch toughness in the AF 1410 steel. The upper shelf energy absorption will decrease in a semi-parabolic relationship as sulfur is progressively increased, Figure 8. Similar behavior exists for a wide range of strength levels in structural steels, References 14, 17, and 18. At low sulfur levels (0.001-0.002%), oxygen contents in excess of 20 ppm substantially reduce the absorbed energy required for ductile rupture in AF 1410 steel, Figure 8. Above 0.005% sulfur, the effect of high oxygen content on notch toughness was seen to diminish.

Microfractography demonstrates an increase of low energy/quasi-cleavage areas in the fracture topography with increasing impurity element content, Figure 9. Apparently these low energy ligaments are associated with crack formation initiating at the second phase particle/matrix interface in areas where the larger inclusions exist, Figure 10.

Limited macrosegregation was present in the VIM/ESR rolled plate product as compared to plate produced from other conventionally processed VIM or VAR ingots.The reduction of such segregation may be inherent in the ESR melting process since melting is restricted to a narrow zone.

The as-quenched microstructure of the VIM/ESR 14Co-10Ni-2Cr-1Mo-0.16C alloy steel (AF 1410) consists of dislocated lath martensite which is oriented in a packet structure, Figure 11. Aging at 950°F (810.0°C) revealed larger lath packets and grain size than normally encountered with the thermal treatment of this steel. Stressing of low carbon steels with a coarse packet size may cause the packets to act as a single grain (Reference 19), thus partially explaining the higher 0.2 pct offset yield strength of the ESR processed steel, particularly in the as-quenched condition.

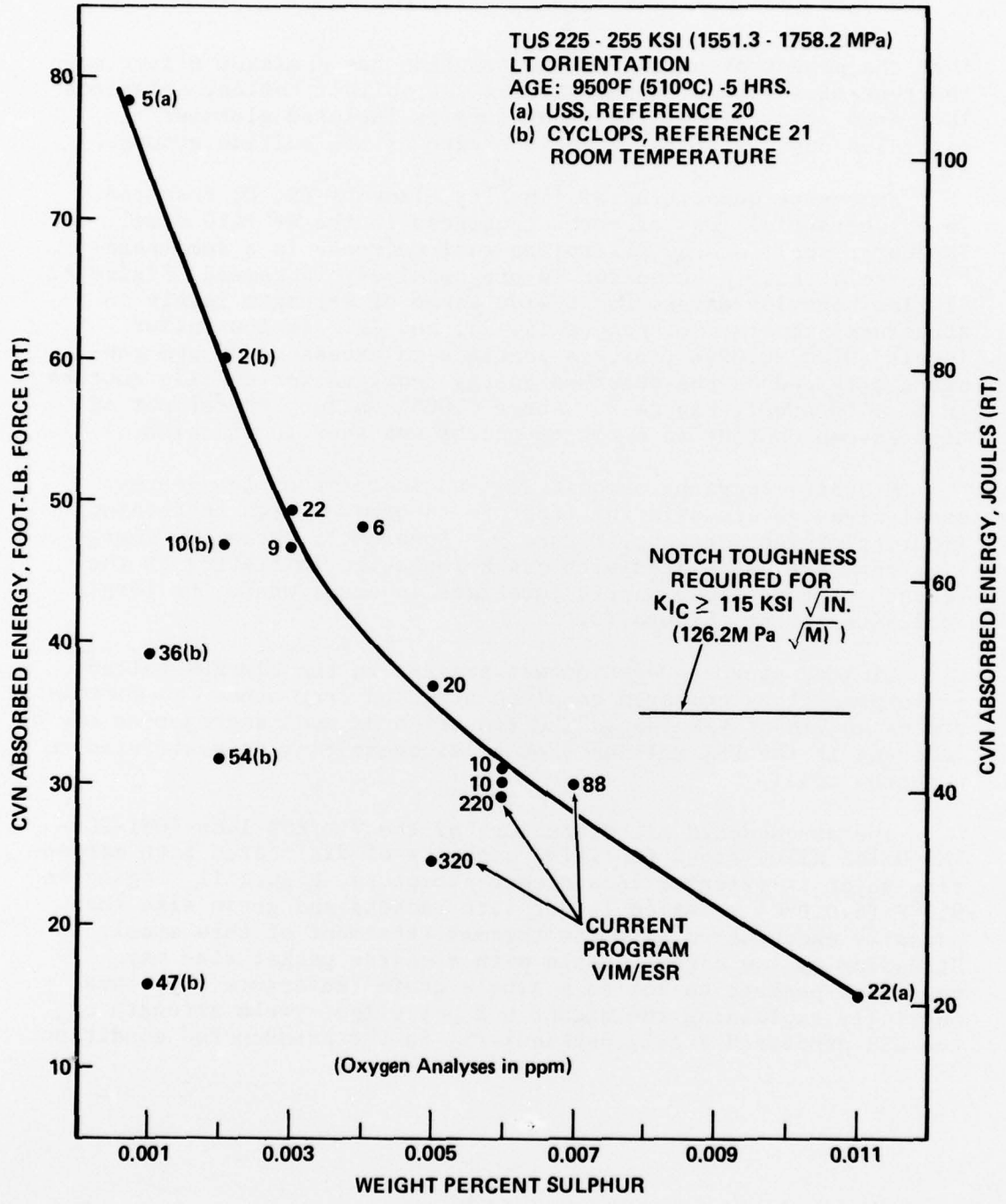
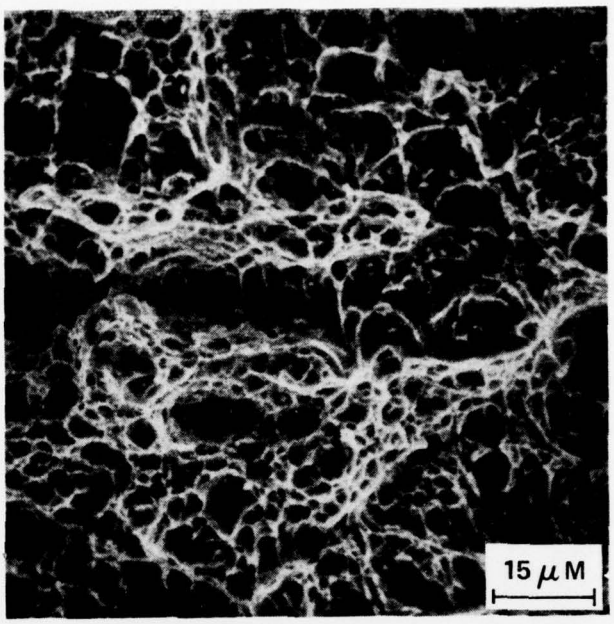
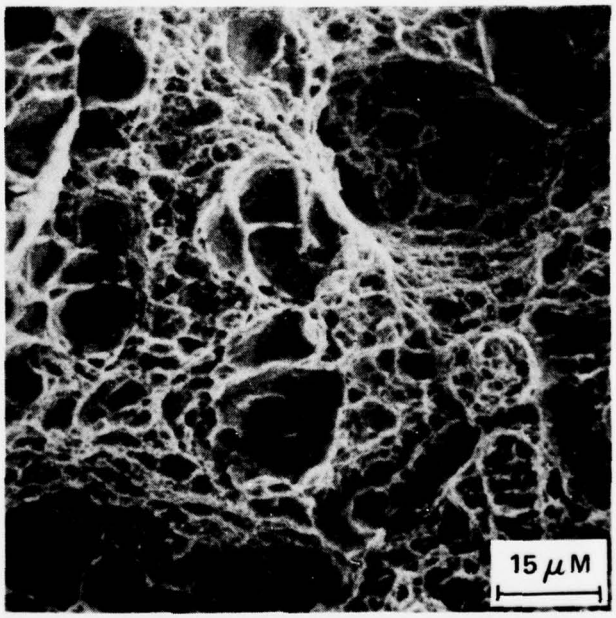


Figure 8 Effect of Sulphur and Oxygen Content on the Notch Toughness of AF 1410 Steel



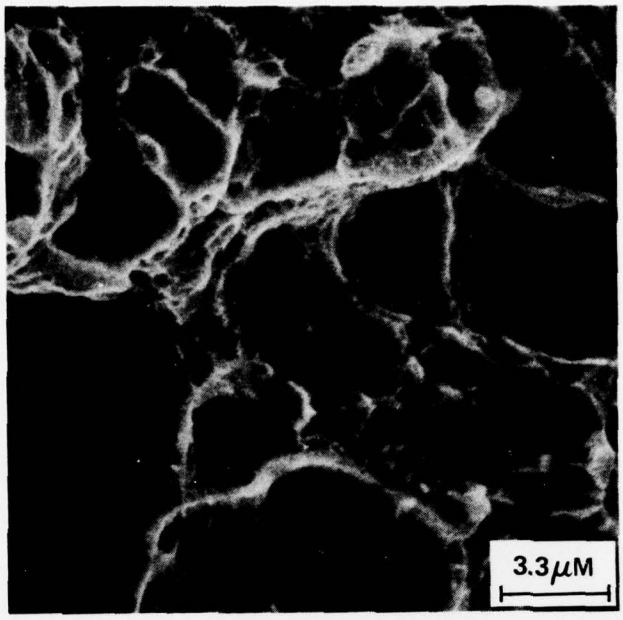
HEAT E
CVN – ABSORBED ENERGY – 31.9 FOOT-LB. FORCE (43.3J)
S – 0.007%, 0 - 88 ppm

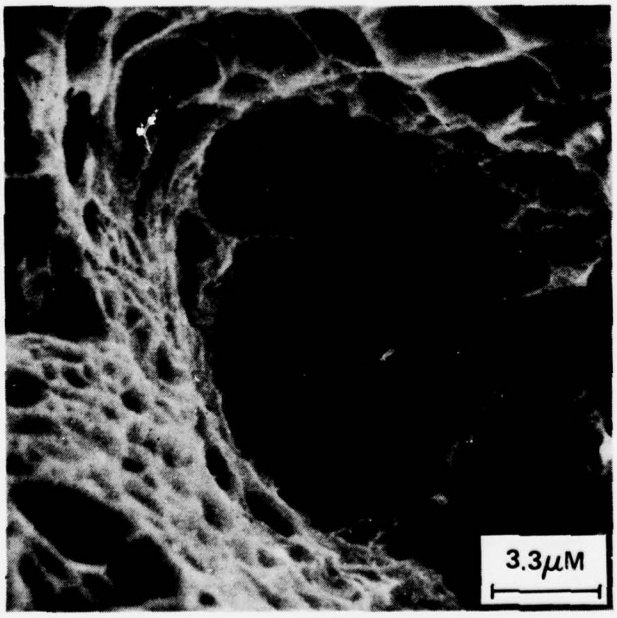


HEAT A
CVN – ABSORBED ENERGY – 25.2 FOOT-LB. FORCE (34.2J)
S – 0.005%, 0 - 320 ppm

AGE: 950°F (510.0°C)/5 HRS. – WQ

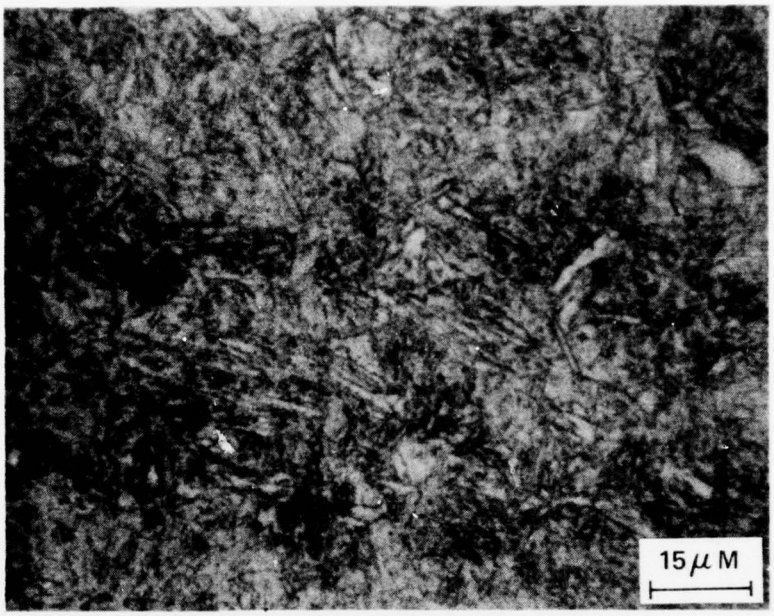
Figure 9 Representative Microfractographs of AF 1410 VIM/ESR Processed Steel



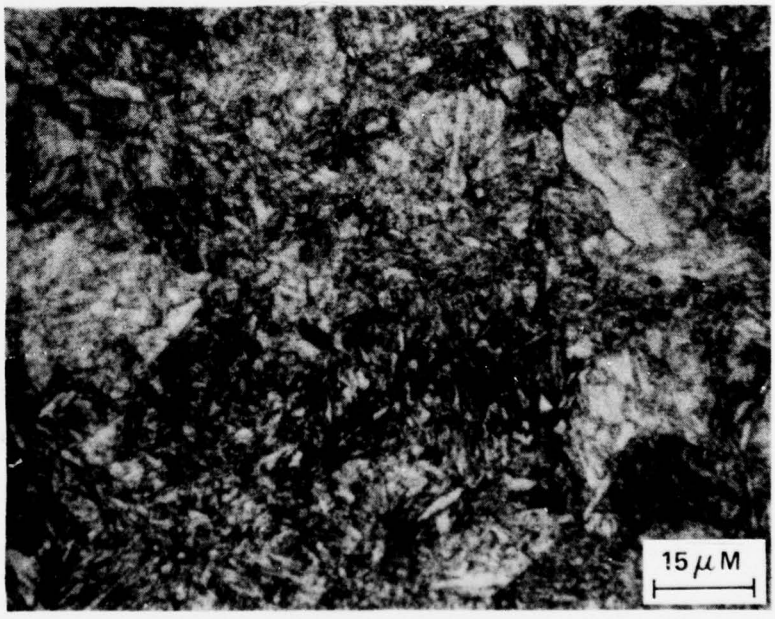


HEAT A S – 0.005%, 0-320 ppm
AGE: 950°F (510.0°C)/5 HRS. – WQ

Figure 10 Effect on Non-Metallic Inclusions Influencing Fracture Processes



A/Q: 1650°F (898.8°C) – WQ/1500°F (815.6°C) – WQ



A/Q + AGED: 950°F (510.0°C) – WQ

Figure 11 Photomicrographs of VIM/ESR AF 1410 Steel in the As-Quenched and A/Q – Aged Condition

C O N C L U S I O N S

1. At a 950°F (810.0°C) aging temperature, the VIM/ESR processed AF 1410 steel plate exceeded a TUS of 230 Ksi (1585.6 MPa) but due to the higher than normal impurity levels did not meet the CVN absorbed energy, $\geq$35 ft-lb (47.4 J) required for a $K_{Ic} \geq 115$ Ksi $\sqrt{in}$ (126.3 MPa $\sqrt{m}$).

2. Electroslag remelting of the AF 1410 steel resulted in an increased inclusion content, thus resulting in considerable toughness degradation. The CaF_2-Al_2O_3-CaO-MgO slag composition did not appreciably decrease the sulfur content of the VIM electrodes. The oxygen increased from levels concomitant with VIM melting to 76-350 ppm thus indicating the need for additional slag and/or metal deoxidation practice.

3. Electron microprobe analysis of the VIM/ESR steel disclosed that the non-metallic inclusions were primarily manganese and chromium containing oxides.

R E C O M M E N D A T I O N S

1. Strong deoxidizing elements should be added continuously to the slag composition during ESR processing to maintain a low oxygen level. Additions of deoxidizing elements to the molten steel, in sufficient quantities to control the oxygen content, would not be expected to result in the desired ductile fracture properties.

2. Additional study of slag compositions and ESR process parameters are required to maximize the desulphurization of the AF 1410 steel alloy.

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