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EVALUATION OF THE MECHANICAL PROPERTIES OF ELECTROSLAG REFINED Fe-12Ni ALLOYS

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16. Abstract

Three Fe-12Ni alloys, individually alloyed with small amounts of V, Ti, and Al, were manufactured through different melting techniques, with special emphasis on electroslag remelting, in order to achieve different levels of metal purity and associated costs. The relative effectiveness of these melting techniques was evaluated from tensile and slow bend fracture toughness behavior at 25°C and -196°C after tempering the test specimens at various temperatures.

This study has shown that the best melting procedure was vacuum induction melting (VIM) with or without electroslag remelting (ESR). VIM+ESR is the recommended procedure since ESR provides increased yield of plate product, a reduction of overall manufacturing costs and, depending on the alloy composition, improved tensile and fracture toughness properties.

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FOREWORD

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SUMMARY

Three Fe-12Ni alloys, individually alloyed with small amounts of V, Ti, and Al, were manufactured through different melting techniques, with special emphasis on electroslag remelting, in order to achieve different levels of metal purity and associated costs. The relative effectiveness of these melting techniques was evaluated from tensile and slow bend fracture toughness behavior at 25°C and -196°C after tempering the test specimens at various temperatures.

This study has shown that the best melting procedure was vacuum induction melting (VIM) with or without electroslag remelting (ESR). VIM+ESR is the recommended procedure since ESR provides increased yield of plate product, a reduction of overall manufacturing costs and, depending on the alloy composition, improved tensile and fracture toughness properties. ESR improved the fracture toughness of the airmelted (AIM) grade of the Fe-12Ni-2V alloy. ESR also raised the ductility of the AIM grade to levels approaching those of the VIM and VIM+ESR grades of the same composition. The method of melting had only a minor effect on the tensile behavior of Fe-12Ni-2V alloy tested at 25°C and -196°C. ESR was highly effective in raising the yield and tensile strengths of the Fe-12Ni-0.2Ti alloy, but ESR did not improve the fracture toughness of this alloy. Good toughness/strength combinations at -196°C are available in all three of the Fe-12Ni alloys tested. For cryogenic applications the Fe-12Ni-0.5Al alloy, based on its manufacturing ease, is a prime candidate for further development.

1.0 INTRODUCTION

The purpose of this program is to evaluate the effects of purity on certain mechanical properties of iron-12-weight-percent-nickel (Fe-12Ni) alloys containing individual small additions of titanium (Ti), vanadium (V) and aluminum (Al). Purity in the alloys used is achieved either through the use of high purity ingredients or through refinement of the melt by vacuum processing or electroslag remelting.

This program is part of an effort to develop and characterize materials useful to NASA for advanced space and aeronautical applications. The efforts are specifically directed toward cost reduction and improvements of fracture toughness of iron-base alloys for low temperature and cryogenic services.

The Fe-12Ni-0.2Ti alloy and the Fe-12Ni-0.5Al alloy materials were prepared in a vacuum induction furnace. The Fe-12Ni-2V alloy ingots were made both in air-induction (AIM) and vacuum-induction (VIM) furnaces. Electroslag remelting (ESR) of all three alloy compositions was done primarily to reduce the size of inclusions, improve the chemical homogeneity and structural features of the cast ingot.

The mechanical property characterization was conducted on plates 28.7 mm (1.13 inch) thick, prepared from each heat of experimental material. Tensile and fracture toughness behavior of seven alloy plates representing the three alloy compositions and the various melting, purification and solidification procedures were examined. Test specimens of each plate were given different heat treatments. Tensile and three-point bend fracture toughness tests were performed to assess the influence of material purity and heat treatment on the mechanical properties of Fe-12Ni alloys at -196°C and 25°C.

2.0 EXPERIMENTAL MATERIALS MANUFACTURE

2.1 Low Cost Approach to Experimental Fe-12Ni Alloys

The experimental materials requirements of this program were small and therefore, laboratory type melting equipment was used to manufacture the three alloy compositions. The high purity Fe-12Ni alloys were prepared in a vacuum-induction furnace using high purity alloying ingredients. The approach used to reduce manufacturing cost of the final plate product is through yield improvement by electroslag remelting.

Other approaches used for the manufacture of low-cost experimental Fe-12Ni alloys included air-induction melting of alloy ingredients of low reactivity, followed by electroslag remelting of the air melted material.

In commercial practice, the Fe-12Ni alloys can be cost-effectively manufactured by techniques other than those described above. These approaches are discussed in a later section of this report.

The stringent chemical analyses requirements of the three experimental Fe-12Ni alloys are as shown in Table I. Preliminary efforts to use low-cost charge materials, such as armco iron, in vacuum-induction furnace to produce the Fe-12Ni-0.2Ti and Fe-12Ni-2V alloys were quite unsuccessful. The control of carbon, titanium and even vanadium proved to be a challenging task during the preparation of the experimental alloys both in the vacuum-induction and air-induction melting systems. The first three vacuum-induction melted heats and two air-induction melted heats of the two Fe-12Ni alloys had to be scrapped because of non-achievement of the required uniform chemical composition.

High purity ingredients were, therefore, used in the subsequent efforts. The basic Fe-12Ni-0.2Ti alloy composition could be made only in the vacuum melting system using high purity charge materials. The Fe-12Ni-2V alloy, however, could be made in both air-induction and vacuum-induction melting systems using only the high purity charge materials. In the case of the Fe-12Ni-0.5Al alloy, the producer preference was to melt it in the vacuum-induction furnace because of anticipated difficulties in achieving the required level of aluminum content in an air-induction furnace. The 60 ppm limit for the content of each residual element in the experimental alloys, placed severe restrictions on the use of scrap and similar low cost charge materials.

2.2 Manufacturing Procedure of Experimental Alloys

Virgin charge materials were used for the manufacture of all Fe-12Ni alloys evaluated in this program. The chemical composition, source and cost information relative to these charge materials are as provided in Table II.

A newly rammed and cured furnace lining of magnesia was used to avoid melt contamination from previous heats. The curing of the furnace lining was done by melting a wash heat of Fe-12Ni alloys prior to processing of each experimental Fe-12Ni alloy heat.

The chemical analyses of the five vacuum-induction melted and two air-induction melted experimental Fe-12Ni alloys are given in Table III. Each melt was cast into a cast iron mold having a 178 mm round cornered, square (RCS) top opening which tapered to 152 mm RCS along a length of 610 mm. The mold was fitted with a special refractory hot top and when filled with molten metal, provided an ingot weighing approximately 160 Kg. The weight of the metal in the hot top was about 28 Kg.

Ingots VE 949, VE 950 and AW 620 were forged into 125 mm thick, 305 mm wide slabs. Ingots VE 965, VE 966, VF-39 and AW 606 were forged and rough machined into 102 mm diameter bars. The ingot hot tops were cut off during the forging operation. Prior to forging, all ingots were heated to 1100°C and soaked at this temperature for a period of one hour.

The slabs were dressed by grinding off the scale from all its surfaces. Each slab was ultrasonically inspected and indicated absence of any internal defects. Subsequently, all slabs were heated to 1100°C, soaked for one hour, and rolled into 29 mm thick, 305 mm wide plates.

The bars produced from vacuum melts VE 965, VE 966, VF-39 and airmelt AW 606 were used as consumable electrodes and electroslag remelted into 152 mm diameter, approximately 55 Kg ingots. The slag used for remelting the Fe-12Ni-0.5Al alloy contained 70% CaF_2 and 30% Al_2O_3; the slag used for the other alloys contained 70% CaF_2, 15% Al_2O_3 and 15% CaO. The electroslag melt in each case was initiated by charging molten slag which was prepared in a graphite crucible in an induction furnace. Electroslag remelting was conducted in a closed mold under an argon atmosphere.

The ESR ingots were dressed by grinding off the ripples on their surface. The ingots were heated to 1100°C and soaked for at least one hour prior to hammer forging into slabs. The end of the slab representing the bottom of the ESR ingot was hot sheared to remove the starter plate plus at least 60 mm section of the ESR ingot adjacent to the starter plate. The slab was reheated to 1100°C and straight away rolled into a plate of the required dimensions.

The plates produced from fully inspected slabs were rechecked by ultrasonic inspection. Normal production mill standards were applied and the plates were judged to be acceptable.

Chemical analysis rechecks were made on ESR plates and the data are as provided in Table III.

3.0 CHARACTERIZATION OF EXPERIMENTAL Fe-12Ni ALLOYS

The Fe-12Ni alloys characterization was limited to the determination of tensile and fracture toughness at room temperature and -196°C of plate specimens in the as-rolled and several annealed (heat treated) conditions. Examination of the microstructures for cleanliness, i.e., sizes, amounts and distribution of various inclusions, stability of austenite, and the microstructural species developed as a consequence of the heat treatments provided to the experimental alloy samples was also conducted.

3.1 Preparation of Test Specimens

Specimen blanks were extracted from each alloy plate by sectioning with an abrasive wheel using copious amount of coolant to avoid burning of the specimen edges. Test specimens were oriented longitudinally, to the principal rolling direction. The tensile test blanks were 152.4 mm long, 25.4 mm wide, 29 mm thick (6" x 1" x 1.13") and the fracture toughness test bars - 215.9 mm long, 50.8 mm wide, 29 mm thick (8 1/2" x 2" x 1.13").

These test specimen blanks were divided into groups. One group of specimen blanks was given no heat treatment which represented material in the "as hot-rolled" condition. The other groups were tempered at 500°, 550°, 600°, 685° or 820°C for two hours followed by water quenching. Only one tensile and one fracture toughness specimen was prepared for each condition of test.

Following heat treatment, the tensile test specimens were machined conforming to ASTM Standard E-8-69. The tensile specimens, tested at room temperature (25°C) and at -196°C, had gage length diameters of 12.8 mm (0.505 in.) and 8.9 mm (0.352 in.) respectively.

The three-point bend fracture toughness test specimens were machined to the geometry and dimensions as indicated in Figure 1. Each specimen was fatigue cracked to an initial crack length to specimen width ratio (a/W) of approximately 0.6.

3.2 Tensile Properties Evaluation

Tensile testing was conducted in a 534 MN Baldwin Universal Testing Machine. Specimens were loaded at a constant strain rate of 0.004 cm/sec. Tensile properties data for the three Fe-12Ni alloys evaluated at 25°C and at -196°C are presented in Tables IV and V respectively.

3.3 Fracture Toughness Evaluation

The three-point bend fracture test fixture used consisted of a base plate with two adjustable roller support blocks and a semi-cylindrical load rod which was bolted to the crosshead of the tensile test machine. The specimen was symmetrically positioned over the two roller supports with a span of 203.2 mm and the load was applied directly over the ligament containing the crack at a speed of 0.203 to 0.254 mm per minute until fracture.

Load deflection curves were plotted on an X-Y recorder from the outputs of a load cell and a double cantilever clip-in displacement gage which was positioned between a stationary and movable column of the testing machine. The deflection of the bend specimen was sensed by the relative vertical displacement of the two columns.

The load/deflection curve and the measurable crack length in the fractured specimen provide data which can be used to calculate the fracture toughness in terms of equivalent energy (K_{Icd}) and J-integral. The empirical formula used for the equivalent energy toughness is:

$$K_{Icd} = \frac{SP_2 \sqrt{A_1/A_2}\, f\,(a/W)}{BW^{3/2}}$$

where,

A_1 = area under curve to maximum load

A_2 = area under curve to P_2

a = crack length

B = specimen thickness

f (a/W) = K calibration

P_2 = load at any point on linear portion of load/deflection curve

S = span for three-point bending

W = specimen width

The J-integral was estimated by a tentative procedure developed by ASTM Task Group E24:01:09. The J-integral value at maximum load (Jm) was calculated from the following relationship:

$$J_m = \frac{2A}{Bb}$$

where,

A = area under the load/deflection curve up to the maximum load point

B = specimen thickness

b = uncracked ligament (W-a)

The results of fracture toughness tests conducted at 25°C and -196°C on specimens of the three Fe-12Ni alloys are summarized in Tables VI and VII, respectively.

3.4 Metallographic Studies

Metallographic studies of the experimental Fe-12Ni alloys included qualitative examinations of the macrostructures representative of the cast and forged condition, grain size in the various grades of plate material, inclusion size, shape and distribution, and the microstructures developed as a consequence of the heat treatments provided to the mechanical test specimens.

Typical cast and forged condition macrostructures of the Fe-12Ni-0.2Ti and Fe-12Ni-2V alloys are shown in Figure 2. The materials do not show any macro-defects, such as segregation or porosity.

The grain size in the plate materials of the various grades of the three experimental Fe-12Ni alloys was estimated by comparison with ASTM standards. Figure 3 shows grain sizes at 100 X in AIM, AIM+ESR, VIM and VIM+ESR plates of Fe-12Ni-2V alloy. Figure 4 shows grain sizes at 100 X of VIM and VIM+ESR plates of Fe-12Ni-0.2Ti alloy and VIM+ESR plate of Fe-12Ni-0.5Al alloy. Plates of all grades of Fe-12Ni alloys investigated in this program show grain sizes finer than ASTM 7.

Typical inclusions seen in the AIM, AIM+ESR, VIM and VIM+ESR grades of Fe-12Ni-2V alloy plates are depicted in Figure 5. Inclusions observed in the VIM, and VIM+ESR grades of Fe-12Ni-0.2Ti alloy and the Fe-12Ni-0.5Al alloy are as shown in Figure 6. All materials were judged to be relatively clean although the ESR grades appear cleaner.

The microstructures of as-rolled condition and the various tempered condition Fe-12Ni-2V alloy plates of the AIM, AIM+ESR, VIM and VIM+ESR grades are shown in Figures 7 through 10. The samples from the plates representative of the various types of melting and refining conditions were polished and examined on the thickness cross section. The etching solution was prepared by mixing 33 parts nitric acid, 33 parts acetic acid, 33 parts water and 1 part hydrofluoric acid. This etching solution prior to use on the metallographic specimens was further diluted in the proportion 1 part of etchant to 20 parts of distilled water. Microstructures of all plate specimens were examined at 1000 x. The as-hot rolled condition microstructures of all four grades of Fe-12Ni-2V alloy showed mixtures of ferrite and austenite. The specimens tempered for two hours at 550°C and 600°C showed approximately the same structural features as the rolled condition specimens. However, the structure in these samples etches to a darker shade. The specimens tempered at 650°C and 685°C, indicated clear signs of austenite transformation. The structure observed was fine, lamellar and consisted of a mixture of ferrite, martensite and small islands of retained austenite. Tempering at 820°C followed by quenching results in the formation of fully martensitic structure.

Microstructural features in ESR plates of Fe-12Ni-2V alloys appear finer and more uniform compared to those in either AIM or VIM grades.

The microstructures of VIM, VIM+ESR grades of Fe-12Ni-0.2Ti alloy and the VIM+ESR grade of Fe-12Ni-0.5Al alloy for the as-rolled and different heat treated conditions are presented in Figures 11 through 13. The microstructural features developed in these two alloys for similarly melted and heat treated conditions are essentially the same as described for the Fe-12Ni-2V alloy.

4.0 CONSIDERATIONS RELATIVE TO COMMERCIAL PRODUCTION OF LOW COST Fe-12Ni ALLOYS

The selection of steels for economy of manufacture is a topic of major interest to both producers and users. Specialty metals producers have been conscious of the inadequacies of the conventional methods of melting, refining and ingot casting. The user, on the other hand, has had several concerns for material availability, cost, optimum utilization in product design, service performance behavior, product liability, prevention of premature failures.

Resolution of these concerns has led to the realization that the conventional two-slag process of manufacturing steels can no longer be considered an economically optimum production process. Specifically, there is a critical need for improving productivity and overall quality of electric arc furnace melted alloy steels.

The electric arc furnace is an excellent tool for economically melting down the cold charge with simultaneous injection of oxygen and slag forming constituents. However, it is not an efficient tool for refining molten metal because it takes 2 to 4 hours time inside the electric arc furnace as compared to 35 to 70 minutes in separate modern refining vessels.

The "outside" electric arc furnace refining treatments of molten steel may include operations such as:

Vacuum degassing
Argon stirring (AS)
Calcium/magnesium argon blowing (CAB)
Vacuum oxygen decarburization (VOD)
Argon-oxygen decarburization (AOD)
Ladle-furnace process (LF)

The objectives of melt treatment outside the electric arc furnace are to accomplish decarburization, prealloying and final deoxidation, desulfurization, degassing and homogenization of chemical composition and adjusting of teeming temperature and stricter controls of such other process variables.

Vacuum induction melting of alloy steels can be economically justified on the basis of high purity requirement of the final product. However, the normal charge materials used for manufacturing vacuum melted alloys are of considerably higher purity (quality) and higher cost as compared to those used in the electric arc furnace.

Melting and casting under vacuum environment per se does not lead to production of sound ingots. Special controls of the ingot solidification process are necessary to provide defect free, dense ingots for further processing into the required mill products. Specialty metals producers, therefore, have a deep appreciation of the inter-relationships amongst the various metal refining and solidification processing steps and the achievement of various levels of product integrity and costs.

Therefore, many strategies for the low-cost production of engineering alloys can be devised. However, the availability of appropriate metal processing equipment will dictate the optimized processing techniques which may be used for the production of a given alloy.

The suggested processing flow sheet for commercial production of very high purity Fe-12Ni alloys and low-cost Fe-12Ni alloys are as outlined below.

4.1 High Purity Fe-12Ni Alloys

1) Vacuum induction melting of virgin charge materials which would provide a final alloy composition of lower impurity content than the specification maximum.
2) Vacuum casting of the alloy of required chemical composition into rectangular electrodes for consumable remelting by the electroslag remelting process.
3) Electroslag remelting of the electrode into a slab ingot suitable for direct rolling into plate.
4) Rolling of the slab ingot into plate of required dimensions.

4.2 Low Cost Fe-12Ni Alloys

The low cost approach to the manufacture of Fe-12Ni alloys could include the following steps dependent upon the type of molten metal secondary refining equipment available to the specialty alloys producer:

1) Electric arc furnace melting of charge consisting of low cost iron, such as armco iron, nickel sinter or pellets, and other alloying constituents of the required chemical purity.
2) Refining of the molten metal in an AOD or VOD vessel, and the preparation of the molten metal of the required final chemical composition.
3) Casting of the molten alloy into rectangular electrodes for electroslag remelting.
4) Electroslag remelting of the electrode into a slab ingot suitable for direct rolling into plate.
5) Rolling of the slab ingot into plate of required dimensions.

If the alloy manufacturer does not have electroslag remelting equipment suitable for producing slab ingots, the alternative is to cast round ESR ingots. These ingots can then be forged into slabs suitable for rolling into plates. This forging step, however, will add a significant cost to the final plate product.

Application of the principles of thermodynamics and kinetics to conventional steelmaking processes has been a very useful tool in understanding, controlling and accomplishing metallurgical reactions. These same principles are applicable to vacuum induction melting, vacuum refining and refining of the molten metal through injection of gases, slag constituents and reactive metal powders.

The main objective of vacuum-induction melting is to produce metals and alloys relatively free from gases and volatile impurities. This objective is attained by: (1) careful selection of charge materials, (2) purification of the melt under vacuum, (3) preventing contamination of the melt during melting and pouring into ingot molds.

Charge materials for vacuum melting are usually virgin metals of high purity or revert vacuum melted scrap.

Purification in vacuum induction melting is achieved through dissociation, deoxidation, degassification and volatilization.

Dissociation of oxides and nitrides is promoted at high temperatures and/or low pressures. The reduction of dissolved oxygen in vacuum induction melting is attained by (1) carbon reduction, (2) hydrogen reduction, and (3) adding a strong oxide-forming element. The first two of these methods yield gaseous reaction products which are pumped away continuously. The third procedure leads to the formation of insoluble oxides which tend to remain in the metal as nonmetallic inclusions.

For the manufacture of very low carbon Fe-12Ni alloys, the most important reactions are metal-deoxidation and reduction of carbon to the desired low levels. This is easily accomplished during vacuum induction melting through carbon deoxidation which produces CO gas as the reaction product according to the following reaction:

$$\underline{C} + \underline{O} = CO\ (gas); \quad K = \frac{P_{CO}}{a_C \cdot a_O}$$

where,

P_{CO} = partial pressure of CO

a_C = activity of carbon

a_O = activity of oxygen

The rate of carbon deoxidation reaction is very fast at steel melting temperatures. The degree of completion and kinetics of reactions occurring in vacuum induction melting can be studied by a monitoring mass spectrometer in combination with samples of chemical analyses and oxygen potential measuring probes. For the preparation of Fe-12Ni alloys, once the molten charge is properly deoxidized, the alloying of the iron-nickel melt with titanium, vanadium, aluminum can be achieved successfully. However, undesirable reactions between the added alloy metals and the crucible refractories are problems which need careful control. An example of this is the addition of titanium to Fe-12Ni alloy. Brief descriptions of the AOD and VOD processes are given below.

The AOD process is being used by the steel industry predominantly for the economical production of stainless steels. Less well known are the applications of the AOD process for the production of super alloys, tool steels, and even chromium free nickel-iron alloys.

The molten metal charge for the AOD refining vessel is usually premelted in either an electric arc furnace or an induction furnace with essentially all the alloying elements required to make a specific alloy composition. The carbon content of the melt is maintained in the range 0.8 to 1.5 percent. The melt is tapped into a ladle, deslagged and then transferred into the AOD vessel. Argon and oxygen in the required proportions are injected through the tuyers located at the bottom back wall of the vessel. The proportion of argon and oxygen are varied from 1:3 at the beginning of the treatment to 3:1 at the end of the treatment period. The duration of the refining period lasts from 1 1/2 to 2 hours depending upon the initial carbon content of the melt. The metal temperature is allowed to rise to 1740°C towards the end of the decarburization period for an effective removal of carbon down to 0.020 percent or even lower. Argon promotes the carbon-oxygen reaction through the removal of carbon monoxide formed during this reaction.

After the carbon is reduced to the required level, a reducing lime-silicon slag is added and the bath stirred with argon only. The excess heat in the metal is adequate to melt the slag. The chemical analysis of the alloy steel is checked and adjusted with the necessary finishing additions.

In the VOD processes, the molten metal containing carbon content less than 0.5 percent and 0.2 percent silicon is deslagged and tapped into a specially designed ladle at a temperature around 1650°C. The ladle is then placed into a sealed vacuum chamber. The chamber is evacuated using a steam ejector pumping system. The ladle has a porous plug at the bottom through which argon is injected for stirring the molten metal. For vacuum decarburization, oxygen is injected below the metal surface through an oxygen lance. The carbon-oxygen reaction supplies heat adequate to compensate the heat lost through radiation, conduction and that which is carried away by the evacuated gases.

Following the decarburization treatment, reductants are added to recover the chromium from the slag back into the metal. Corrective additions are made to adjust the alloy composition.

The ladle is then taken out of the vacuum chamber and the metal is cast into ingot molds in the conventional manner.

The solidification defects occurring in conventionally cast ingots can be significantly reduced through the use of the electroslag process of metal refining and ingot solidification. Consumable electrode processes, such as ESR, VAR (vacuum arc remelting) are electrically less efficient than primary melting processes, e.g., electric arc or induction melting because in the former processes, melting and ingot solidification is done in water-cooled molds. An important consideration in ESR is the achievement of improved yield and properties that may result in energy and labor savings in the manufacture of mill products capable of meeting specific requirements.

The capability of producing slabs and other shaped ingots, besides rounds, high productivity, high yield, and improved surface quality and ingot structure, are important benefits of the ESR process relative to low-cost manufacture of Fe-12Ni alloys.

4.1 Fe-12Ni Alloys Plate Manufacturing Cost Estimates

Estimates of comparative costs of preparing Fe-12Ni alloy plates using either high purity or low-cost charge materials are as given below:

A. Charge Materials

Material	Costs $/Kg
Iron (Glidden)	1.28
Armco iron	0.39
Nickel pellets	4.85

Titanium	3.68
Vanadium (80% Fe-V)	13.23
Aluminum (shots or pigs)	1.08

B. Vacuum Induction Melting System (VIM)

10 ton production system and plant - $4.2 M
Annual production - 8200 tons

Annual Operating Costs (in thousands)

Depreciation	520
Maintenance	160
Labor	240
Overhead	360
Power	400
Other Utilities	100
Miscellaneous	70
	$1,850

Cost/Ton VIM Metal - approximately $226

C. Electric Arc Furnace and Argon Oxygen Decarburization (AOD) or Vacuum Oxygen Decarburization (VOD) Unit

Electric Arc Furnace (EF) - 20 ton

AOD or VOD Unit - 25 ton

EF + AOD or VOD Systems and Plant Cost $5.6 M

Annual Production - 30,000 tons

Annual Operating Costs (in thousands)

Depreciation	560
Maintenance	200
Labor	400
Overhead	600
Power	110
Other Utilities	60
Miscellaneous	150
	$2,080

Cost/Ton of EF + AOD or VOD Steel - approximately $70

D. ESR Plant - Slab and Round Ingots

Plant Cost	$2.0 M
Annual Production	5000 tons

Annual Operating Costs (in thousands)

Depreciation	200
Maintenance	100
Labor	140
Overhead	210
Power	30
Other Utilities	20
Slag and Consumables	140
Miscellaneous	60
	$900

Cost/Ton ESR ingot - approximately $180

E. Manufacturing Cost Estimates of Fe-12Ni Alloy Plates

1. Fe-12Ni-2V Alloy

Manufacturing Steps	VIM + ESR Route		EF+AOD (or VOD)+ESR Route	
	Processing Step Cost	Cumulative Cost	Processing Step Cost	Cumulative Cost
Charge Materials, late additions and mark-up costs	-	$2572/T*	-	$1644/T
Furnace Charge		$2572/1000Kg		1644/1000Kg
Primary Melting	$226/T	2798/1000Kg	$70/T	1714/1000Kg
Electrode Casting	50/T	2848/950Kg	44/T	1758/940Kg
Electrode Preparation	20/T	2868/940Kg	25/T	1783/925Kg
ESR (round)	180/T	3048/925Kg	180/T	1963/910Kg
ESR (slab)	180/T	3048/915Kg	180/T	1963/900Kg
Forge ESR round	331/T	3379/900Kg	331/T	2294/885Kg
Roll ESR slab	440/T	3488/915Kg	440/T	2403/900Kg
Roll Forged Slab	440/T	3819/900Kg	440/T	2734/885Kg
Cost of Plate - ESR Slab		$3.81/Kg		$2.67/Kg
Cost of Plate - ESR Round		$4.24/Kg		$3.09/Kg

*Ton - Metric ton (1000 Kg)

2. Fe-12Ni-0.2Ti Alloy

Manufacturing Steps	VIM + ESR Route		EF+AOD(or VOD)+ESR Route	
	Processing Step Cost	Cumulative Cost	Processing Step Cost	Cumulative Cost
Charge materials, late additions and mark-up costs	-	$2267/T	-	$1295/T
Furnace Charge	-	$2267/1000Kg	-	$1295/1000Kg
Primary Melting	$226/T	2493/1000Kg	$ 70/T	1365/1000Kg
Electrode Casting	50/T	2543/1000Kg	44/T	1409/940Kg
Electrode Preparation	20/T	2563/940Kg	25/T	1434/925Kg
ESR (round)	180/T	2743/925Kg	180/T	1614/910Kg
ESR (slab)	180/T	2743/915Kg	180/T	1614/900Kg
Forge ESR round	331/T	3074/900Kg	331/T	1945/885Kg
Roll ESR slab	440/T	3183/915Kg	440/T	2054/900Kg
Roll Forged ESR slab	440/T	3514/900Kg	440/T	2385/885Kg
Cost of Plate - ESR Slab		$3.48/Kg		$2.28/Kg
Cost of Plate - ESR Round		$3.90/Kg		$2.69/Kg

3. Fe-12Ni-0.5Al Alloy

Manufacturing Steps	VIM + ESR Route		EF+AOD(or VOD)+ESR Route	
	Processing Step Cost	Cumulative Cost	Processing Step Cost	Cumulative Cost
Charge materials, late additions and mark-up costs	-	$2254/T	-	$1280/T
Furnace Charge	-	$2254/1000Kg	-	$1280/1000Kg
Primary Melting	$226/T	2480/1000Kg	$ 70/T	1350/1000Kg
Electrode Casting	50/T	2530/950Kg	44/T	1394/940Kg
Electrode Preparation	20/T	2550/940Kg	25/T	1419/925Kg
ESR (round)	180/T	2730/925Kg	180/T	1599/910Kg
ESR (slab)	180/T	2730/915Kg	180/T	1599/900Kg
Forge ESR round	331/T	3061/900Kg	331/T	1930/885Kg
Roll ESR slab	440/T	3170/915Kg	440/T	2039/900Kg
Roll Forged ESR slab	440/T	3501/900Kg	440/T	2370/885Kg
Cost of Plate - ESR Slab		$3.46/Kg		$2.27/Kg
Cost of Plate - ESR Round		$3.89/Kg		$2.68/Kg

5.0 DISCUSSION

5.1 Manufacturing Considerations of Fe-12Ni Alloys

The present study has indicated that the vanadium added Fe-12Ni alloy can be manufactured satisfactorily by both air melting and vacuum melting techniques. It can also be readily processed through electroslag remelting. However, the vanadium units of this alloy carry a high price tag. When primary melting of this alloy is done in the vacuum induction melting unit, followed by electroslag remelting, the basic cost of the alloy becomes highest of all three compositions investigated. In commercial practice, the Fe-12Ni-2V alloy can be advantageously manufactured via the three step, electric arc furnace melting, AOD or VOD refining followed by electroslag remelting of a slab ingot. This procedure will lead to lowest-cost attainable for the final mill product.

The titanium added Fe-12Ni alloy will require primary melting in a vacuum induction furnace. The high reactivity of titanium makes its addition in the air melting furnace a very difficult task. A compromise would be to melt the basic alloy in the electric arc furnace and make the titanium addition in the VOD unit. However, during casting of the electrodes of this alloy, titanium loss is possible. Also, during electroslag remelting further titanium loss could occur. One procedure in commercial manufacturing practice is to add an excess amount of titanium to the primary melt to compensate for its loss during electroslag remelting. But regardless of the procedures used, titanium control in this alloy composition is a complex task. Another problem which requires mentioning is revert melting of this alloy in the electric arc furnace. The titanium oxidized during melting of this alloy scrap in the electric arc or air induction furnace will severely contaminate the melt with TiO_2 inclusions. A safer practice, therefore, is to prepare this alloy composition in the vacuum induction furnace using mostly virgin charge materials and small amounts of mill revert scrap. The manufacturing cost of this alloy could be high if VOD refining unit is not available for alloying the basic melt prepared in an air furnace.

The aluminum added Fe-12Ni alloy is the easiest to melt, both in the primary melting and electroslag remelting systems. In commercial manufacturing practice, Fe-12Ni-0.5Al alloy can be primary melted using the electric arc furnace and the AOD or VOD refining units. Electroslag remelting is facilitated by the fact that Al_2O_3 (alumina) is a component of the slag used in this process.

From the viewpoints of manufacturing ease and economies, the aluminum added Fe-12Ni alloy would be the primary choice with the titanium added alloy as the secondary choice for further materials development and characterization considerations.

A summary of manufacturing cost estimates of commercially producing Fe-12Ni alloy plates of the three experimental compositions of a high-purity grade and a low-cost grade is as follows:

Alloy		Plate Manufacturing Cost Estimate	
		High Purity Grade	Low-Cost Grade
Fe-12Ni-0.2Ti	from ESR Slab	- $3.48/Kg	$2.28/Kg
	from ESR Round	- $3.90/Kg	$2.69/Kg
Fe-12Ni-2V	from ESR Slab	- $3.81/Kg	$2.67/Kg
	from ESR Round	- $4.24/Kg	$3.09/Kg
Fe-12Ni-0.5Al	from ESR Slab	- $3.46/Kg	$2.27/Kg
	from ESR Round	- $3.89/Kg	$2.68/Kg

5.2 Tensile Behavior

5.2.1 Yield Strength, Tensile Strength and Ductility at 25°C

To facilitate comparative study of the tensile behavior of the three Fe-12Ni alloys, the data for the tensile tests conducted at 25°C are presented in a bar chart form in Figure 14. An examination of this chart shows that the room temperature yield strength of the Fe-12Ni-2V alloy ranged from 586 MPa to 855 MPa (85 to 124 Ksi) for the various heat treated conditions with minor differences among the AIM, AIM+ESR, VIM and VIM+ESR grades. The yield strength was highest generally for the hot rolled condition in all grades. The 685°C tempering treatment of this alloy provided the lowest yield strength ranging from 586 MPa to 634 MPa (85 to 92 Ksi).

The tensile strength of the Fe-12Ni-2V alloy ranged from 738 MPa to 1117 MPa (107 to 162 Ksi). The AIM and AIM+ESR grades provided higher tensile strength than the VIM and VIM+ESR grades. It is presumed that the higher carbon content in the AIM grade may be responsible for the development of higher tensile properties.

The ductility, as reflected by the area reduction (R/A) values for the various grades of Fe-12Ni-2V alloy, followed the normal trend that higher R/A values are associated with low yield strength values and lower R/A values associated with high yield strength values. The 685°C tempering treatment of this alloy, compared to tempering at other temperatures, developed highest ductility. ESR did not affect the tensile properties of the Fe-12Ni-2V alloy, but it improved the ductility of the AIM grade, approaching the ductility values attained by the VIM and VIM+ESR grades of this composition.

The VIM grade of Fe-12Ni-0.2Ti alloy, as noted in Figure 14, developed yield strengths ranging from 586 MPa to 662 MPa (85 to 96 Ksi); whereas, the VIM+ESR grade of the same composition provided improved yield strength - 703 MPa to 813 MPa (102 to 118 Ksi) for the hot rolled and different heat treated conditions. The higher ductility of the VIM grade of Fe-12Ni-0.2Ti alloy compared to the VIM+ESR grade for identical heat treatments can be explained on the basis of the lower yield and tensile strength developed for the former grade.

The VIM+ESR grades of Fe-12Ni-0.5Al alloy provided yield strengths (Ref. Figure 14) from 655 MPa to 800 MPa (95 to 116 Ksi) and tensile strengths of 765 MPa to 1055 MPa (111 to 153 Ksi) for the various heat treatments.

5.2.2 Yield Strength, Tensile Strength and Ductility at -196°C

Figure 15 presents yield strength and ductility comparisons for the three experimental Fe-12Ni alloy plates from different melt procedures and heat treated conditions which were tested at -196°C.

The yield strength of different grades of Fe-12Ni-2V alloy varied from 924 MPa to 1145 MPa (134 to 166 Ksi) and the corresponding tensile strength, from 1089 MPa to 1476 MPa for the various heat treated conditions. The ductility of the vanadium added alloy for the AIM and AIM+ESR grade was slightly lower (R/A values of 49% to 61%) than that of the VIM and VIM+ESR grades (R/A values of 56% to 68%).

The yield strength of the VIM and VIM+ESR processed Fe-12Ni-0.2Ti alloy plates for the different heat treatments ranged from 889 MPa to 1255 MPa (129 to 182 Ksi) and the tensile strength, from 1089 MPa to 1407 MPa (158 to 204 Ksi). The area reduction (R/A) values corresponding to these strength levels are in the range 61% to 68%.

The yield and tensile strength values for the VIM+ESR processed Fe-12Ni-0.5Al alloy are in the ranges 855 MPa to 1179 MPa (124 to 171 Ksi), and 979 MPa to 1379 MPa (142 to 200 Ksi) respectively. The reduction of area values are in the 62% to 68% range for the various heat treated conditions.

The yield and tensile strength values of the titanium and aluminum added Fe-12Ni alloys show a drop when tempered at 550°C to 600°C and recover in the tempering range 685°C to 820°C.

The preceding discussions of the tensile behavior of the different grades of Fe-12Ni alloys at 25°C and -196°C lead to the following observations:

- The method of melting had only a minor effect on the room temperature and -196°C tensile behavior of the Fe-12Ni-2V alloy for the various tempered conditions.
- VIM+ESR processed Fe-12Ni-0.2Ti alloy provided significantly higher yield and tensile strengths compared to the VIM melted same alloy tempered at various temperatures and tested at 25°C and -196°C.
- The three Fe-12Ni alloys, melted by the VIM+ESR process, generally displayed their highest yield strengths for tempering temperatures of 685°C or 820°C.
- All three Fe-12Ni alloys displayed high ductility even at the cryogenic temperature of -196°C with area reduction (R/A) values above 50%.

5.3 Fracture Behavior

The fracture toughness of the experimental Fe-12Ni alloys evaluated at 25°C and -196°C and as represented by K_{Icd} and J_m parameter values from the three point bend pre-cracked test specimens are presented in Tables VI and VII respectively.

Figures 16 and 17 present a comparative assessment of the three Fe-12Ni alloys tested respectively at 25°C and -196°C.

5.3.1 Fracture Toughness at 25°C

The AIM and AIM+ESR grades of the Fe-12Ni-2V alloy heat treated at the same temperatures provided fracture toughness K_{Icd} values ranging from 127 MPa$\sqrt{m}$ to 259 MPa$\sqrt{m}$ (114 Ksi$\sqrt{in}$ to 236 Ksi$\sqrt{in}$) respectively.

For the VIM and VIM+ESR grades of the above alloy, the K_{Icd} values for similar heat treated conditions ranged from 196 MPa$\sqrt{m}$ to 277 MPa$\sqrt{m}$ (178 Ksi$\sqrt{in}$ to 252 Ksi$\sqrt{in}$) and 191 MPa$\sqrt{m}$ to 302 MPa$\sqrt{m}$ (174 Ksi$\sqrt{in}$ to 275 Ksi$\sqrt{in}$) respectively.

These data indicate that ESR improved the fracture toughness of the Fe-12Ni-2V alloy. The VIM grade of this alloy provided higher K_{Icd} values compared to the AIM grade. Also, the VIM+ESR grade of Fe-12Ni-2V alloy displayed higher fracture toughness than the AIM+ESR grade.

The VIM and VIM+ESR grades of the Fe-12Ni-0.2Ti alloy, heat treated at various temperatures, provided fracture toughness K_{Icd} values respectively in the ranges 192 MPa$\sqrt{m}$ to 262 MPa$\sqrt{m}$ (174 Ksi$\sqrt{in}$ to 238 Ksi$\sqrt{in}$) and 192 MPa$\sqrt{m}$ to 230 MPa$\sqrt{m}$ (175 Ksi$\sqrt{in}$ to 209 Ksi$\sqrt{in}$). These data indicate that ESR provided no improvement in the fracture toughness of VIM grade of Fe-12Ni-0.2Ti alloy.

Similarly, fracture toughness K_{Icd} values of the VIM+ESR grade of the Fe-12Ni-0.5Al alloy varied from 186 MPa$\sqrt{m}$ to 216 MPa$\sqrt{m}$ (161 Ksi$\sqrt{in}$ to 197 Ksi$\sqrt{in}$). Compared to the VIM+ESR grade of Fe-12Ni-0.2Ti alloy, the VIM+ESR grade of the Fe-12Ni-0.5Al alloy displayed slightly lower K_{Icd} fracture toughness values.

5.3.2 Fracture Toughness at -196°C

The fracture toughness data of the Fe-12Ni alloys tested at -196°C are comparatively presented in Figure 17.

The AIM and AIM+ESR grades of the Fe-12Ni-2V alloy provided fracture toughness values respectively in the ranges from 78 MPa$\sqrt{m}$ to 133 MPa$\sqrt{m}$ (71 Ksi$\sqrt{in}$ to 121 Ksi$\sqrt{in}$) and from 77 MPa$\sqrt{m}$ to 190 MPa$\sqrt{m}$ (70 Ksi$\sqrt{in}$ to 173 Ksi$\sqrt{in}$). Therefore, ESR improved the fracture toughness of the AIM grade of this alloy.

In contrast, the VIM+ESR grade of the Fe-12Ni-2V alloy displayed poorer fracture toughness, with K_{Icd} values ranging from 38 MPa$\sqrt{m}$ to 153 MPa$\sqrt{m}$ (35 Ksi$\sqrt{in}$ to 139 Ksi$\sqrt{in}$) compared to the VIM grade whose K_{Icd} values ranged from 148 MPa$\sqrt{m}$ to 263 MPa$\sqrt{m}$ (132 Ksi$\sqrt{in}$ to 239 Ksi$\sqrt{in}$).

Thus, in regard to the fracture behavior of the various melt grades of Fe-12Ni-2V alloy, at -196°C the VIM grade provided the highest fracture toughness values.

The fracture toughness, K_{Icd} values of the VIM and VIM+ESR grades of the Fe-12Ni-0.2Ti varied from 187 MPa$\sqrt{m}$ to 256 MPa$\sqrt{m}$ (170 Ksi$\sqrt{in}$ to 233 Ksi$\sqrt{in}$) and from 148 MPa$\sqrt{m}$ to 258 MPa$\sqrt{m}$ (135 Ksi$\sqrt{in}$ to 235 Ksi$\sqrt{in}$) respectively. Therefore, ESR did not improve the fracture toughness of the VIM grade.

Comparing the VIM+ESR grades of the Ti and Al added Fe-12Ni alloys, it is observed that the former alloy developed high values of fracture toughness (over 200 MPa$\sqrt{m}$) when tempered at 685°C and 820°C and the latter alloy developed similar high K_{Icd} values when tempered at 500°C, 550°C and 600°C.

5.3.3 J_m Parameter Values

In Figures 16 and 17, the bottom bar charts provide data comparing the J_m values for the three Fe-12Ni alloys for the different heat treatments and tested respectively at 25°C and -196°C.

The 25°C J_m parameter values for the AIM and VIM grades of the Fe-12Ni-2V alloy indicate markedly higher toughness of the latter grade. ESR improved the toughness of the AIM grade but not of the VIM grade.

Concerning the Fe-12Ni-0.2Ti alloy, the 25°C J_m parameter values of the VIM grade are significantly higher than the VIM+ESR grade.

The J_m parameter values of the VIM+ESR grade of the Fe-12Ni-0.5Al tested at 25°C are higher than those of the VIM+ESR grade of the Fe-12Ni-0.2Ti alloy.

The J_m parameter values of the Fe-12Ni-2V alloy for tests conducted at -196°C are in the range 0.01 to 0.15 MJ/m^2. Exceptions are the AIM+ESR grade of this alloy tempered at 600°C and 650°C and the VIM grade tempered at 685°C and 820°C.

The Fe-12Ni-0.2Ti alloy of both VIM and VIM+ESR grades provided J_m values higher than 0.14 MJ/m^2. The J_m values of the similar grade of Fe-12Ni-0.5Al are significantly higher than those of the titanium added Fe-12Ni alloy.

The significance of high J_m parameter values of the VIM melts of all three Fe-12Ni alloys and VIM+ESR melts of the titanium and aluminum added Fe-12Ni alloys may be explained by their higher plasticity associated with lower gaseous impurity and non-metallic inclusion contents. There is a good correlation between the exceptional cleanliness of the microstructure of the VIM+ESR grade of Fe-12Ni-0.5Al alloy and its high J_m values for tests conducted at -196°C.

5.4 Microstructures

Evaluations of microstructures of the three Fe-12Ni alloy plates of different melt grades which were tempered at different temperatures have led to the following observations:

- All plates displayed fine grain size, smaller than ASTM No. 7 (Figures 3 and 4).
- The VIM grades showed a large number of tiny non-metallic inclusions (Figures 5(c) and 6(a)). The AIM grade of the Fe-12Ni-2V alloy showed lower inclusion content (Figure 5(a)) than its VIM counterpart. However, electroslag remelted grade plates of Fe-12Ni alloys were cleaner than VIM and AIM grades.
- The VIM+ESR grade of Fe-12Ni-0.5Al alloy plates showed the smallest size and the least number of non-metallic inclusions of the three Fe-12Ni compositions prepared by the various melting techniques.
- Microstructures developed in three Fe-12Ni alloy plates were unaffected by the melting practices used. The microstructural features observed are consistent with findings reported in NASA Technical Note D-8232 (May 1976) concerning the development of various Fe-12Ni alloys.

6.0 CONCLUSIONS

Three Fe-12Ni alloys, individually alloyed with small amounts of V, Ti, and Al, were manufactured through different melting techniques, with particular emphasis on electroslag remelting, in order to achieve different levels of metal purity and associated costs. The relative effectiveness of these melting techniques was determined from tensile and slow bend fracture toughness behavior evaluated at 25°C and -196°C after tempering of the test specimens at various temperatures. Conclusions of this study are as follows:

- The best melting procedure was VIM with or without ESR. VIM+ESR is the recommended procedure since ESR provides an increased yield of plate product, a reduction of overall manufacturing costs, and, depending on the alloy composition, improved tensile or fracture toughness properties.
- ESR improved the fracture toughness of the AIM grade of the Fe-12Ni-2V alloy. ESR also improved the ductility of the AIM grade to levels approaching those of the VIM and VIM+ESR grades of the same composition. The method of melting had only a minor effect on the tensile behavior of Fe-12Ni-2V alloy tested at 25°C and -196°C.
- ESR was highly effective in raising the yield and tensile strengths of the VIM grade of the Fe-12Ni-0.2Ti alloy. However, ESR did not improve the fracture toughness characteristics of this alloy.

- Good toughness/strength combinations at -196°C are available in all three of the Fe-12Ni alloys tested. For example, the titanium-added Fe-12Ni alloy manufactured by the VIM-ESR technique, and tempered at 685°C, provided at -196°C a maximum yield strength of 1255 MPa (182 Ksi) and a fracture toughness K_{Icd} value of 2.25 MPa$\sqrt{m}$ (205 Ksi$\sqrt{in}$).
- The Fe-12Ni-0.5Al alloy, based on its manufacturing ease by the low-cost EF+AOD (or VOD)+ESR methods and its low temperature toughness/strength properties, is a prime candidate for further development and evaluation as promising material for cryogenic applications.

GENERAL REFERENCES

1. Witzke, W. R., Stephens, J. R. Effect of Minor Relative Metal Additions on Fracture Toughness of Iron-12-Percent-Nickel Alloy at -196°C and 25°C. NASA Technical Note D-8232 (May 1976).

2. Brown, W. F., Stanley, J. E. Plane strain crack toughness testing of high strength metallic materials. ASTM STP 410, pp. 1-65, 1967.

3. Landes, J. D., Begley, J. A. Test results from J-integral studies - an attempt to establish a J_{IC} testing procedure. ASTM STP 560, Fracture Analysis, pp. 170-180, 1974.

TABLE I

EXPERIMENTAL Fe-12Ni ALLOY COMPOSITION

ALLOY	I	II	III
Nominal Designation	Fe-12Ni-.2Ti-.05C	Fe-12Ni-2V	Fe-12Ni-.5 Al
Metallic Elements (percent by weight)			
Nickel	12.0 - 13.0	12.0 - 13.0	12.0 - 13.0
Silicon	0.010 max	0.010 max	0.010 max
Titanium	0.17 - 0.26	-	-
Vanadium	-	1.7 - 2.1	-
Aluminum	-	-	0.25 - 0.50
Iron	Balance	Balance	Balance
Nonmetallic Elements (parts per million by weight)			
Carbon	400 - 600	200 - 800	100 - 500
Nitrogen	50 max	50 max	50 max
Oxygen	100 max	100 max	100 max
Phosphorus	50 max	50 max	50 max
Sulfur	50 max	50 max	50 max

Note: All other elements in above alloys shall not exceed 60 ppm by weight.

TABLE II

CHARGE MATERIALS USED FOR THE MANUFACTURE
OF EXPERIMENTAL Fe-12Ni ALLOYS,
THEIR SOURCE AND COSTS

Iron*	Glidden	A-101-B	Melting Stock
C-0.002,	Si-0.002,	Mn-0.004,	S-0.002
P-<0.001,	Ni-0.014,	Co-0.005,	Fe-99.1
H-0.0015,	O-<0.0552,	N-0.0017,	Cu-0.005
As-<0.0002	Ag-<0.0010	Bi-<0.0001,	Pb-0.0002
Sn-0.002	W-0.001	V-0.003	
			Cost $1.28/Kg

Nickel - Inco Nickel Pellets			
All elements - parts per million (ppm)			
C - <10	Co - <0.2	Cu - <1	Fe - <20
O - 69	N - 7	H - <1	S - 9
Al - <1	Sb - <0.2	As - <0.2	Bi - <0.2
Cd - <0.2	Pb - <0.2	Mn - <1	P - <0.2
Se - <0.2	Si - 2	Ag - <0.2	Te - <0.2
Th - <0.2	Sn - <1	Zn - <0.5	
			Cost $4.85/Kg

Titanium* - C.P. grade - Frankel Company			
Fe - <0.20	Si - <0.01	Mn - <0.01	O_2 - <0.10
Ti - 99.6% minimum			
H - <100 ppm	N - <100 ppm	Bi - <1 ppm	
Pb - <10 ppm	Sb - <10ppm	Ag - <1 ppm	
			Cost $3.68/Kg

*Note: Content of each element in weight percent unless otherwise reported.

TABLE II (Cont'd)

CHARGE MATERIALS USED FOR THE MANUFACTURE
OF EXPERIMENTAL Fe-12Ni ALLOYS,
THEIR SOURCE AND COSTS

Ferro Vanadium* - Grade 80% FeV - Union Carbide Corp.			
V - 77.19	Si - 0.80	C - 0.21	Mn - 0.08
S - 0.012	P - 0.016		
		Cost $13.23/Kg	

Carbon* - No. 18 grade graphite - Schuler Industries		
C - 97.5 to 98.5	S - <0.02	Ash - 0.5 to 0.70
Volatiles - 0.50 to 0.60	Moisture - Approx. 0.50	
		Cost $0.46/Kg

Aluminum* - 3/8" Shot, Reynolds Metals Co.				
Al - 99.87	Si - 0.06	Fe - 0.06	Cu - <0.01	Mn - <0.01
Mg - <0.01	Cr - <0.01	Zn - 0.01	Ni - <0.01	
Pb - <0.01	Sn - <0.01	Ti - <0.01		
			Cost $1.08/Kg	

*Note: Content of each element in weight percent unless otherwise reported.

TABLE III

CHEMICAL ANALYSES OF EXPERIMENTAL
Fe-12Ni ALLOYS

GRADE	VIM					AIM	
Elements	VE 949	VE 950	VE 965	VE 966	VF-39	AW-606	AW-602
Fe	Base	Base	Base	Base	Base	Base	Base
Ni	12.2	12.1	12.0	12.0	12.1	12.65	12.8
Si	0.009	0.005	<0.01	<0.005	<0.010	<0.01	<0.01
Ti	0.19	--	0.18	--	--	--	--
V	--	1.90	--	1.78	--	1.88	1.76
Al	--	--	--	--	0.46	--	--
C	0.057	0.057	0.052	0.047	0.035	0.088	0.073
N	3 PPM	4 PPM	6 PPM	5 PPM	5 PPM	202 PPM	191 PPM
O	27 PPM	22 PPM	69 PPM	36 PPM	7 PPM	90 PPM	77 PPM
P	<0.005	<0.005	<50 PPM	<50 PPM	<0.005	0.005	0.005
S	0.0048	0.003	30 PPM	30 PPM	0.004	<0.005	<0.005
SEMI PRODUCT	SLAB	SLAB	BAR	BAR	BAR	BAR	SLAB
REFINE	--	--	ESR	ESR	ESR	ESR	--
FINAL PRODUCT	29 mm thick plates						

CHEMICAL ANALYSES OF ESR PLATES

ELEMENTS	VE 965	VE 966	VF-39	AW 606
Fe	Base	Base	Base	Base
Ni	12.1	11.8	11.8	12.6
Si	0.03	0.01	0.03	0.02
Ti	0.114	--	--	--
V	--	1.68	--	1.71
Al	--	--	0.39	<0.05
C	0.049	0.046	0.036	0.080
O	118 PPM	62 PPM	64 PPM	81 PPM
N	10 PPM	11PPM	6 PPM	188 PPM

TABLE IV

TENSILE PROPERTIES OF Fe-12Ni ALLOYS AT ROOM TEMPERATURE (25°C)

Alloy & Melting Mode	Tempered and Quench Temperature (2 hours) °C	Ultimate Tensile Strength		Yield Strength (0.2%)		Elongation (50.8 mm gage)	Area Reduction
		MPa	Ksi	MPa	Ksi	%	%
Fe-12Ni-2V (AIM)	Hot Rolled	1103	160	807	117	14	53
	550	883	128	724	105	21	66
	685	800	116	634	92	18	70
	820	938	136	765	111	15	63
Fe-12Ni-2V (AIM+ESR)	Hot Rolled	1117	162	772	112	13	56
	550	855	124	717	104	20	71
	685	807	117	614	89	19	73
	820	945	137	765	111	16	66
Fe-12Ni-2V (VIM)	Hot Rolled	1062	154	855	124	18	65
	550	821	119	696	101	22	73
	685	772	112	634	92	19	76
	820	896	130	745	108	17	73
Fe-12Ni-2V (VIM+ESR)	Hot Rolled	1041	151	696	101	16	64
	550	800	116	676	98	19	73
	685	738	107	586	85	19	76
	820	896	130	710	103	17	68

TABLE IV (Continued)

TENSILE PROPERTIES OF Fe-12Ni ALLOYS AT ROOM TEMPERATURE (25°C)

Alloy & Melting Mode	Tempered and Quench Temperature (2 hours) °C	Ultimate Tensile Strength		Yield Strength (0.2%)		Elongation (50.8 mmgage)	Area Reduction
		MPa	Ksi	MPa	Ksi	%	%
Fe-12Ni-0.2Ti (VIM)	Hot Rolled	841	122	627	91	19	74
	550	745	108	662	96	22	76
	685	724	105	586	85	20	77
	820	758	110	614	89	19	76
Fe-12Ni-0.2Ti (VIM+ESR)	Hot Rolled	903	139	703	102	16	71
	550	855	124	807	117	21	72
	685	1076	156	807	117	16	65
	820	1089	158	813	118	16	67
Fe-12Ni-0.5Al (VIM+ESR)	Hot Rolled	903	131	655	95	18	70
	550	765	111	717	104	25	73
	685	1055	153	800	116	16	66
	820	1027	149	772	112	17	66

TABLE V

TENSILE PROPERTIES OF Fe-12Ni ALLOYS AT LIQUID NITROGEN TEMPERATURE (-196°C)

Alloy & Melting Mode	Tempering Temperature (2 hours) °C	Ultimate Tensile Strength		Yield Strength (0.2%)		Elongation (50.8 mm gage)	Area Reduction
		MPa	Ksi	MPa	Ksi	%	%
Fe-12Ni-2V (AIM)	Hot Rolled	1476	214	1145	166	16	51
	550	1255	184	1041	151	24	55
	685	1200	174	986	143	17	60
	820	1303	189	1117	162	16	53
Fe-12Ni-2V (AIM+ESR)	Hot Rolled	1400	203	1117	162	15	49
	550	1200	174	1027	149	22	58
	685	1145	166	1020	148	19	61
	820	1234	179	965	140	16	51
Fe-12Ni-2V (VIM)	Hot Rolled	1413	205	1069	155	19	59
	550	1214	176	993	144	24	63
	685	1145	166	1034	150	17	64
	820	1227	178	1062	154	21	68
Fe-12Ni-2V (VIM+ESR)	Hot Rolled	1379	200	1089	158	17	58
	550	1186	172	1000	145	19	56
	600	1103	160	945	137	24	63
	650	1089	158	924	134	22	64
	685	1048	152	993	144	14	63
	820	1220	177	1076	156	19	52

TABLE V (Continued)

TENSILE PROPERTIES OF Fe-12Ni ALLOYS AT LIQUID NITROGEN TEMPERATURE (-196°C)

Alloy & Melting Mode	Tempering Temperature (2 hours) °C	Ultimate Tensile Strength		Yield Strength (0.2%)		Elongation (50.8 mm gage)	Area Reduction
		MPa	Ksi	MPa	Ksi	%	%
Fe-12Ni-0.2Ti	Hot Rolled	1220	177	1124	163	20	65
(VIM)	550	1089	158	993	144	25	66
	685	1103	160	889	129	18	64
	820	1110	161	965	140	21	68
Fe-12Ni-0.2Ti	Hot Rolled	1276	185	993	144	18	63
(VIM+ESR)	550	1207	175	993	144	23	63
	600	1289	187	931	135	23	65
	650	1358	197	1000	145	17	61
	685	1407	204	1255	182	17	61
	820	1372	199	1193	173	21	64
Fe-12Ni-0.5Al	Hot Rolled	1220	177	1000	145	23	66
(VIM+ESR)	500	1179	171	855	124	25	64
	550	979	142	883	128	22	68
	600	1089	158	1014	147	23	62
	685	1379	200	1172	170	18	65
	820	1331	193	1179	171	17	64

TABLE VI

FRACTURE TOUGHNESS OF Fe-12Ni ALLOYS TESTED AT ROOM TEMPERATURE (25°C)

Alloy & Melting Mode	Heat Treatment Aging Temperature (2 hours) °C	K_{Icd}^{+} Energy Balance		J_m^*	
		$MPa\sqrt{m}$	$Ksi\sqrt{in}$	$\frac{M\ Joules}{m^2}$	$\frac{in\text{-}lb}{in^2}$
Fe-12Ni-2V (AIM)	Hot Rolled	127	116	0.11	626
	550	199	181	0.27	1570
	685	211	192	0.27	1569
	820	142	129	0.09	497
Fe-12Ni-2V (AIM+ESR)	Hot Rolled	125	114	0.05	287
	550	248	226	0.38	2181
	685	259	236	0.35	1978
	820	179	163	0.22	1279
Fe-12Ni-2V (VIM)	Hot Rolled	196	178	0.26	1469
	550	264	240	0.53	3010
	685	277	252	0.47	2690
	820	214	195	0.37	2116
Fe-12Ni-2V (VIM+ESR)	Hot Rolled	191	174	0.18	1032
	550	263	239	0.38	2154
	685	302	275	0.48	2715
	820	271	247	0.37	2139

$$^{+}K_{Icd} = \frac{SP_2\sqrt{A/A_2}}{BW^{3/2}} f(a/w)$$

$$^{*}J_m = \frac{2A}{bB}$$

TABLE VI (Continued)

FRACTURE TOUGHNESS OF Fe-12Ni ALLOYS TESTED AT ROOM TEMPERATURE (25°C)

Alloy & Melting Mode	Heat Treatment Aging Temperature (2 hours) °C	K_{Icd}+ Energy Balance		J_m*	
		MPa$\sqrt{m}$	Ksi$\sqrt{in}$	M Joules/m^2	in-lb/in^2
Fe-12Ni-0.2Ti (VIM)	Hot Rolled	192	174	0.24	1390
	550	225	205	0.43	2429
	685	262	238	0.41	2365
	820	214	195	0.38	2196
Fe-12Ni-0.2Ti (VIM+ESR)	Hot Rolled	230	209	0.25	1410
	550	220	200	0.23	1334
	685	192	175	0.18	1029
	820	214	195	0.22	1271
Fe-12Ni-0.5Al (VIM+ESR)	Hot Rolled	201	183	0.29	1648
	550	216	197	0.40	2276
	685	186	169	0.22	1265
	820	199	181	0.27	1569

$$+K_{Icd} = \frac{SP_2\sqrt{A_1/A_2}}{BW^{3/2}} f\,(a/w)$$

$$*J_m = \frac{2A}{bB}$$

TABLE VII

FRACTURE TOUGHNESS OF Fe-12Ni ALLOYS TESTED AT LIQUID NITROGEN TEMPERATURE (-196°C)

Alloy & Melting Mode	Heat Treatment Aging Temperature (2 hours) °C	K_{Icd}^{+} Energy Balance		J_m^{*}	
		MPa$\sqrt{m}$	Ksi$\sqrt{in}$	$\frac{M\ Joules}{m^2}$	$\frac{in\text{-}lb}{in^2}$
Fe-12Ni-2V (AIM)	Hot Rolled	96	87	0.05	304
	550	78	71	0.05	265
	685	133	121	0.11	625
	820	126	115	0.09	496
Fe-12Ni-2V (AIM+ESR)	Hot Rolled	77	70	0.01	72
	550	132	120	0.11	638
	600	175	159	0.19	1104
	650	190	173	0.23	1320
	685	130	118	0.12	699
	820	115	105	0.08	439
Fe-12Ni-2V (VIM)	Hot Rolled	162	147	0.12	692
	550	145	132	0.12	700
	685	179	163	0.18	1015
	820	263	239	0.47	2700
Fe-12Ni-2V (VIM+ESR)	Hot Rolled	64	58	0.04	213
	550	38	35	0.01	45
	600	96	89	0.08	443
	650	136	124	0.11	625
	685	153	139	0.14	785
	820	152	138	0.09	541

TABLE VII (Continued)

FRACTURE TOUGHNESS OF Fe-12Ni ALLOYS TESTED AT LIQUID NITROGEN TEMPERATURE (-196°C)

Alloy & Melting Mode	Heat Treatment Aging Temperature (2 hours) °C	K_{Icd}+ Energy Balance		J_m*	
		$MPa\sqrt{m}$	$Ksi\sqrt{in}$	$\frac{M\ Joules}{m^2}$	$\frac{in\text{-}lb}{in^2}$
Fe-12Ni-0.2Ti (VIM)	Hot Rolled	201	183	0.24	1362
	550	187	170	0.17	997
	685	256	233	0.34	1913
	820	226	206	0.29	1668
Fe-12Ni-0.2Ti (VIM+ESR)	Hot Rolled	258	235	0.42	2374
	550	148	135	0.14	818
	600	173	157	0.20	1159
	650	162	147	0.16	905
	685	225	205	0.30	1725
	820	243	221	0.38	2192
Fe-12Ni-0.5Al (VIM+ESR)	Hot Rolled	305	278	0.47	2711
	500	259	236	0.46	2599
	550	245	223	0.33	1912
	600	235	214	0.34	1931
	685	198	180	0.24	1375
	820	181	165	0.15	865

$$+K_{Icd} = \frac{SP_2\sqrt{A_1/A_2}\, f\,(a/w)}{BW^{3/2}}$$

$$*J_m = \frac{2A}{bB}$$

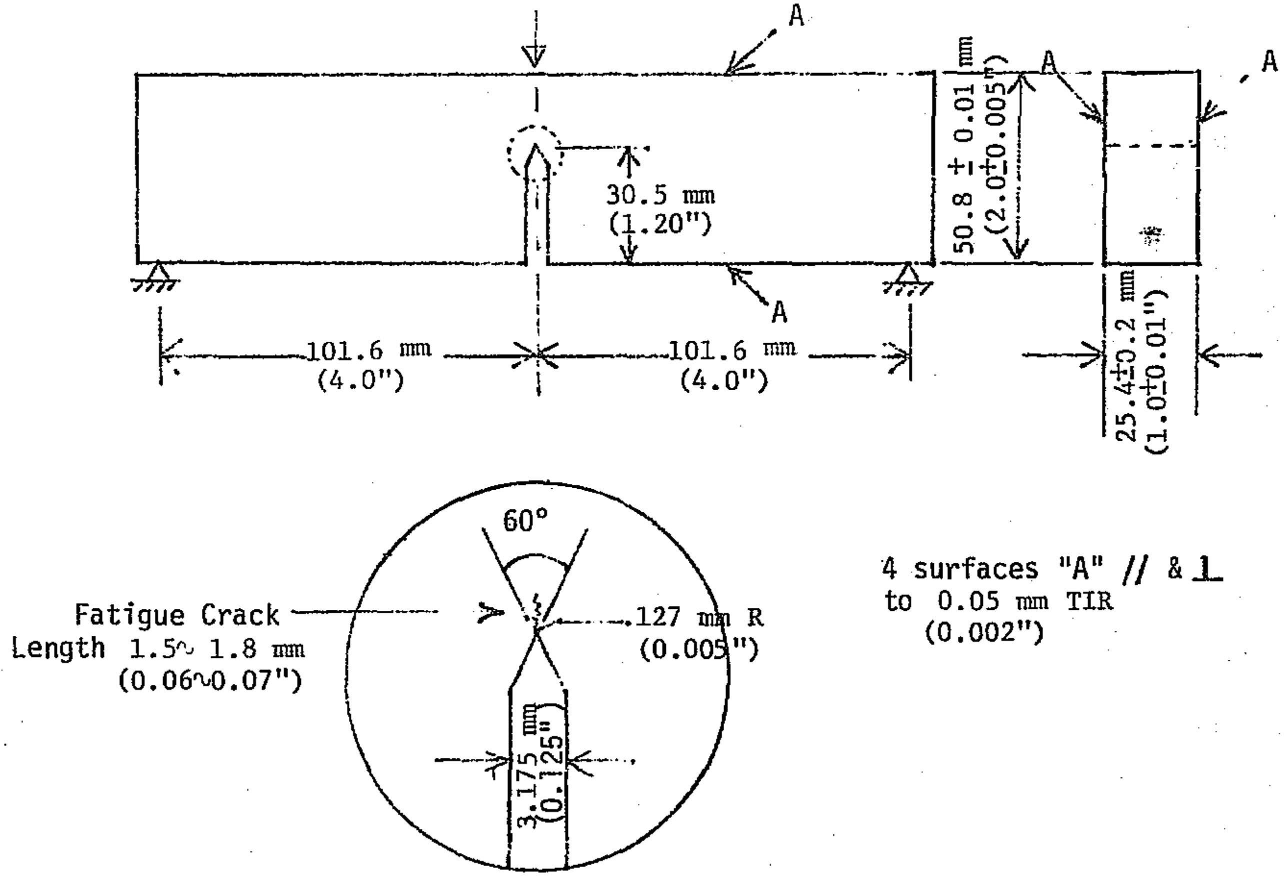
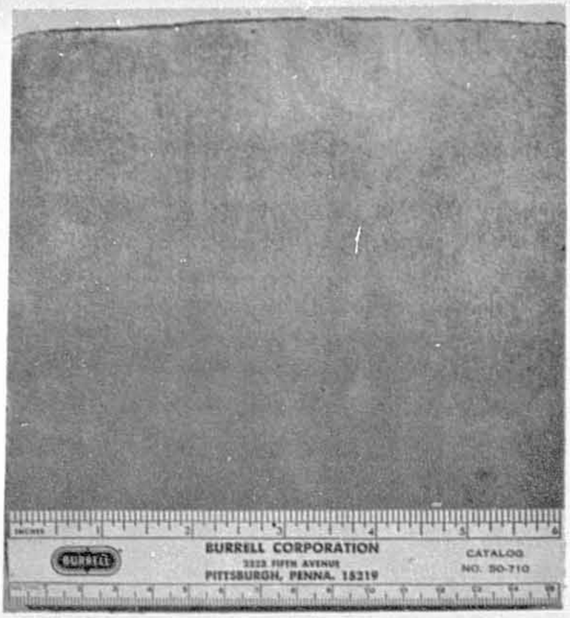
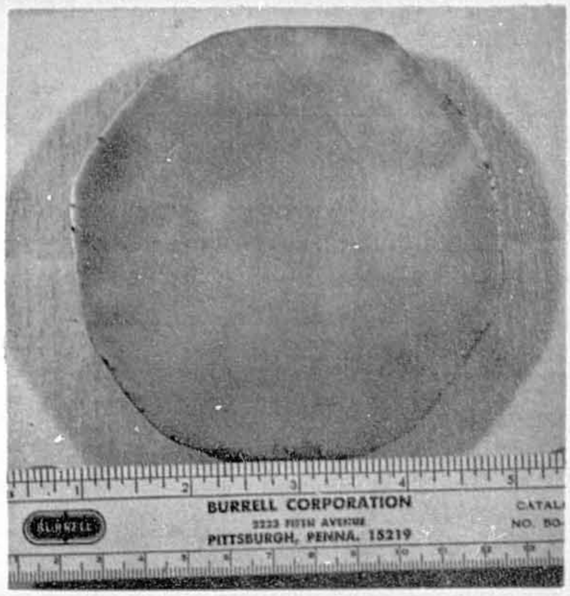


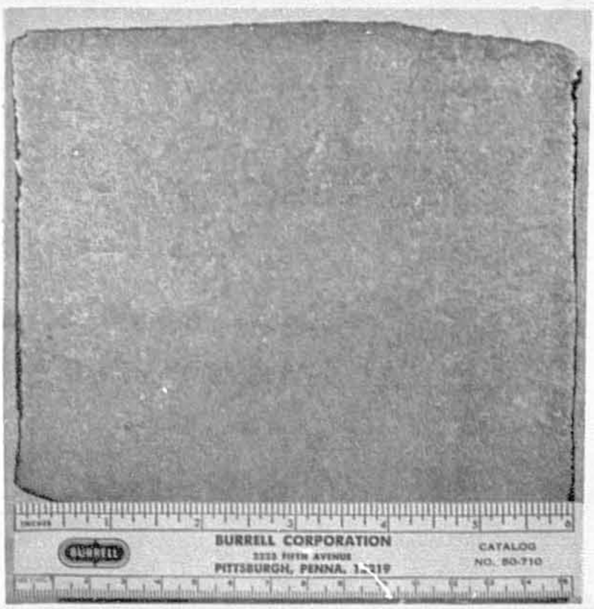
Figure 1. Three Point Bend Fracture Toughness Test Specimen

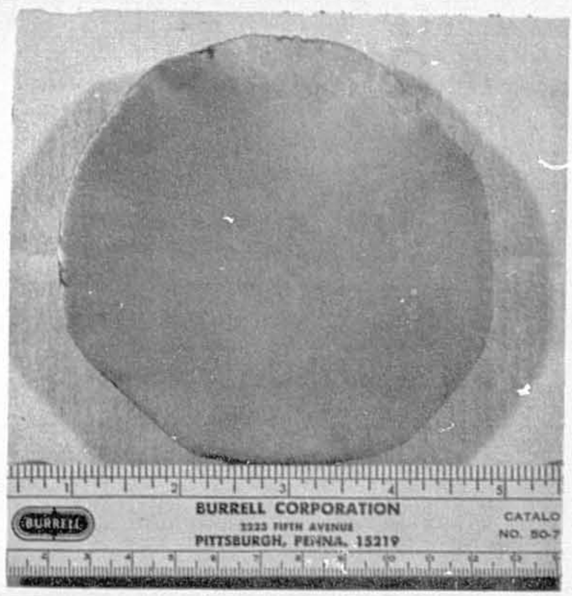




Ingot Forged

Fe-12Ni-0.2Ti alloy

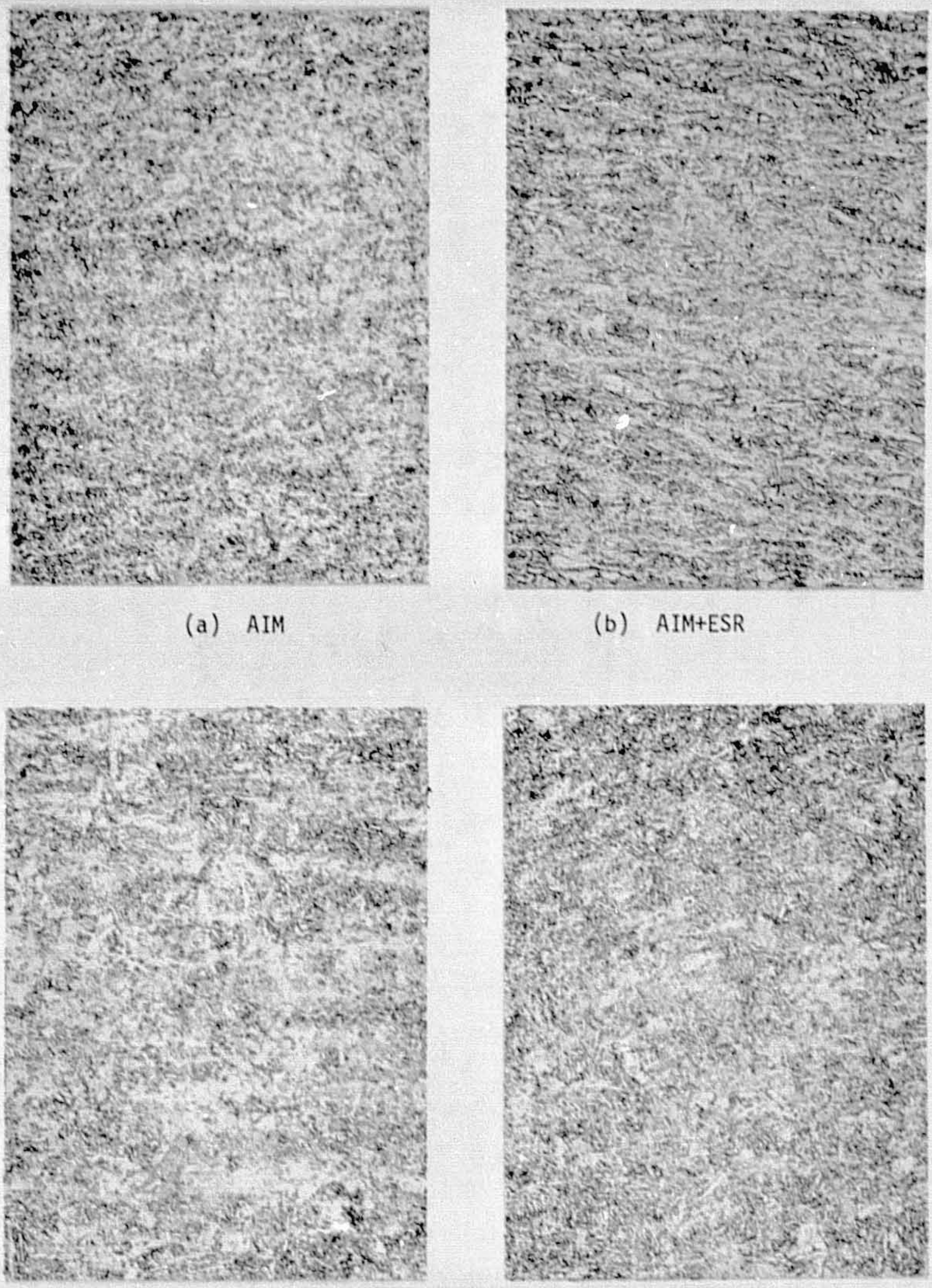




Ingot Forged

Fe-12Ni-2V alloy

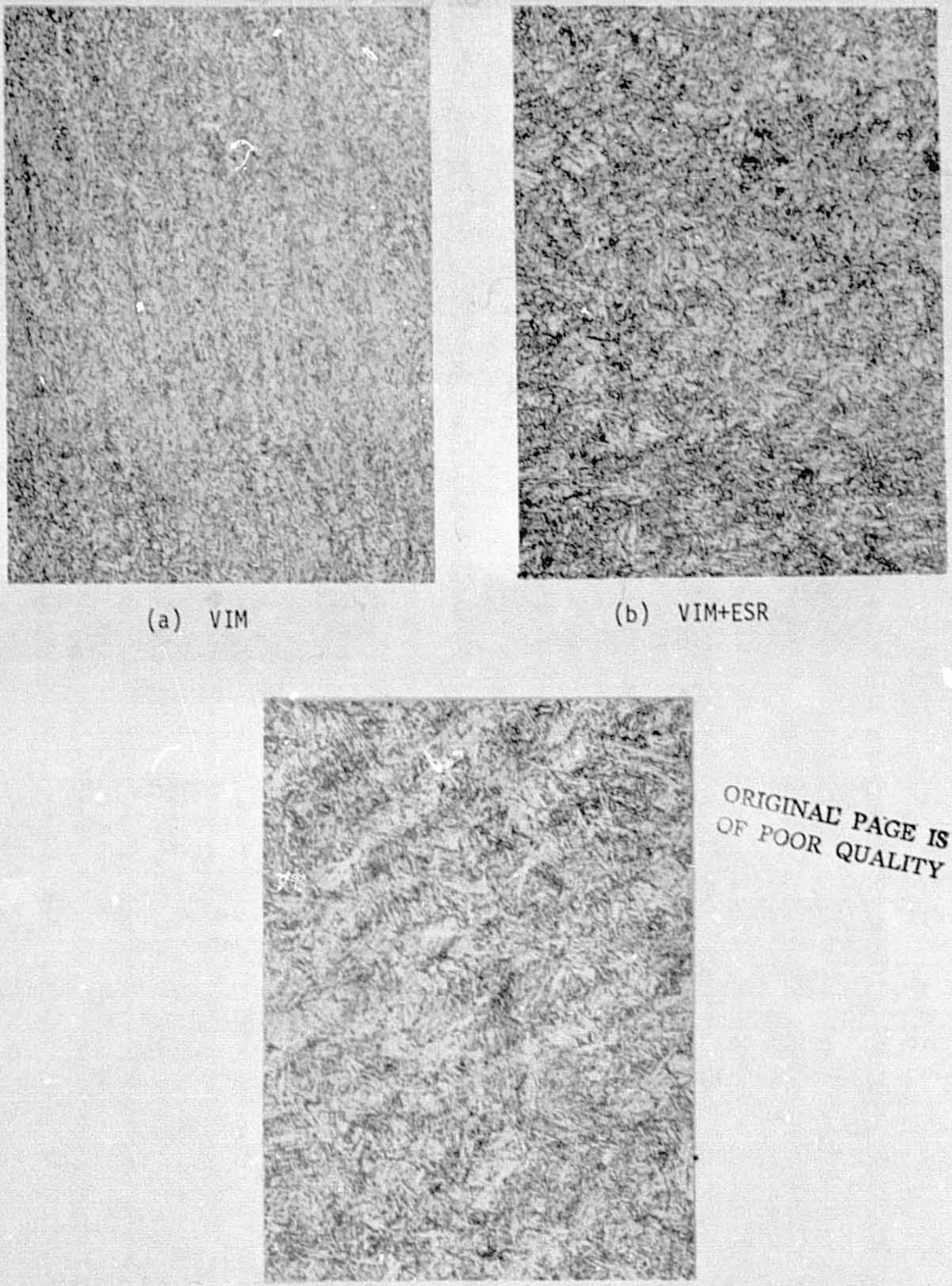
FIGURE 2 . Typical macrostructure of two Fe-12Ni alloys.



(a) AIM (b) AIM+ESR

(c) VIM (d) VIM+ESR

FIGURE 3. Grain size of AIM (a), AIM+ESR (b), VIM (c), VIM+ESR (d) plates of Fe-12Ni-2V alloy.
Magnification x100, hot rolled condition.

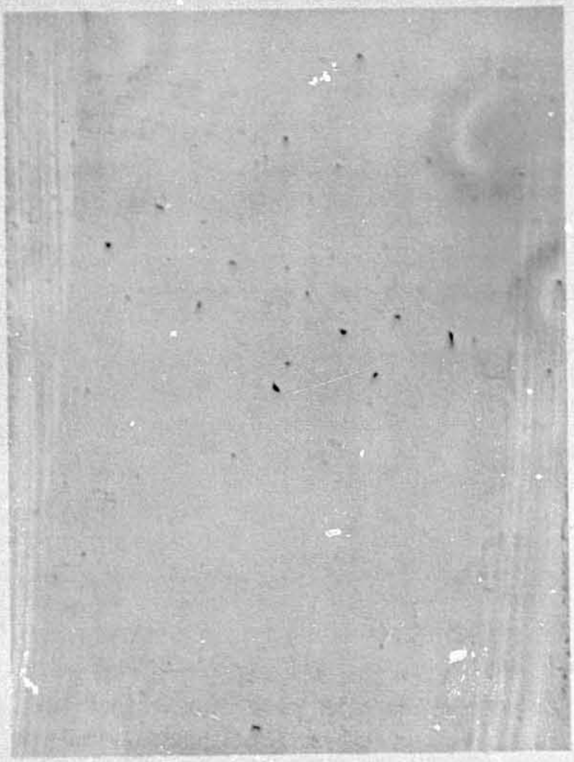


(a) VIM

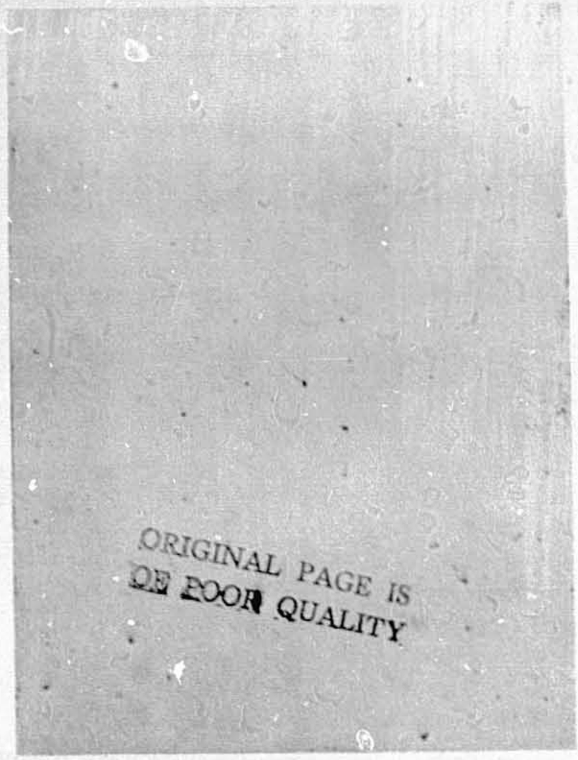
(b) VIM+ESR

(c) VIM+ESR

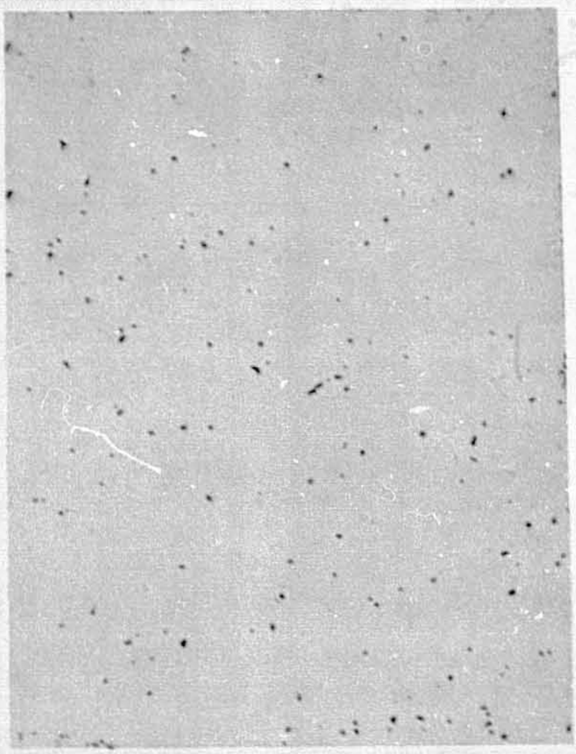
FIGURE 4. Grain size of VIM (a), VIM+ESR (b) plates of Fe-12Ni-0.2Ti alloy and VIM+ESR (c) plates of Fe-12Ni-0.5Al alloy. Magnification x100, hot rolled condition.



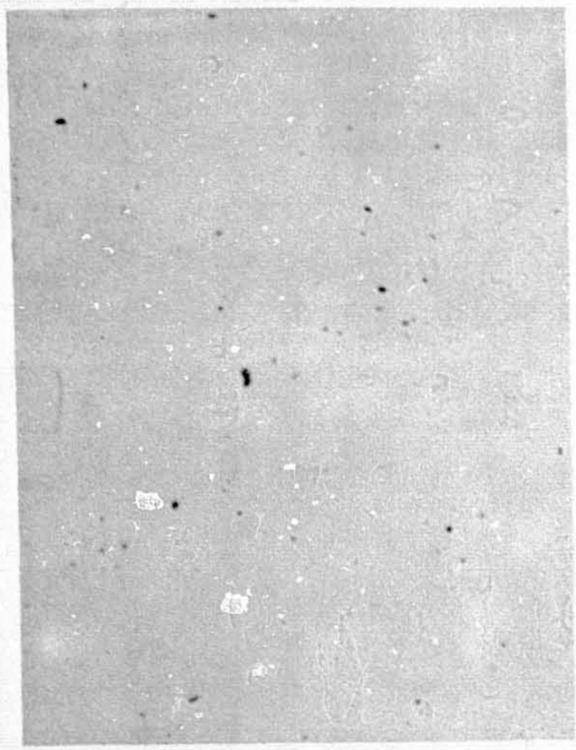
(a) AIM



(b) AIM + ESR

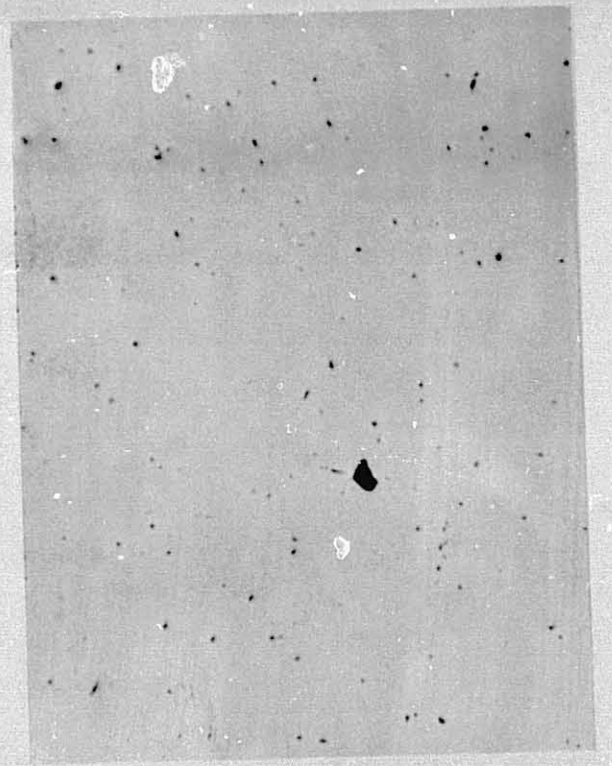


(c) VIM

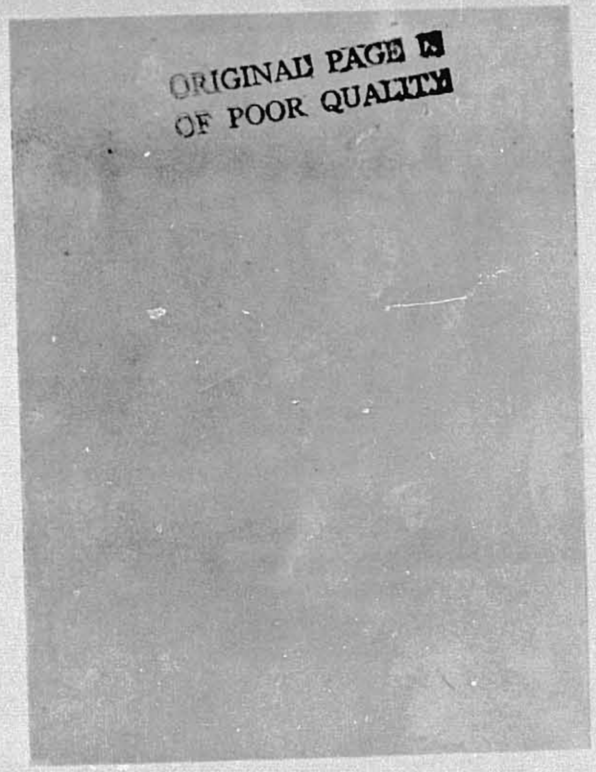


(d) VIM + ESR

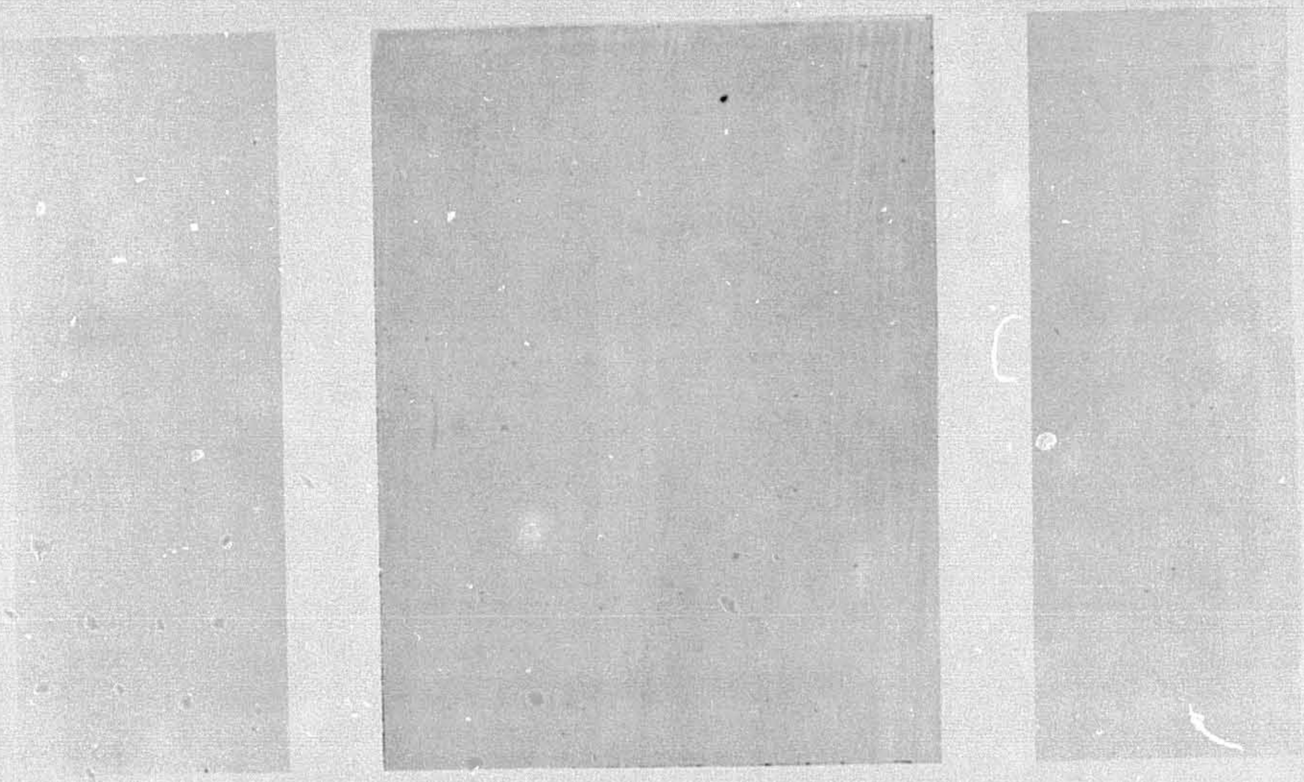
FIGURE 5 . Typical inclusions in AIM, AIM+ESR, VIM, and VIM+ESR grades of Fe-12Ni-2V alloys.
Magnification - x100, as rolled condition.



(a) VIM

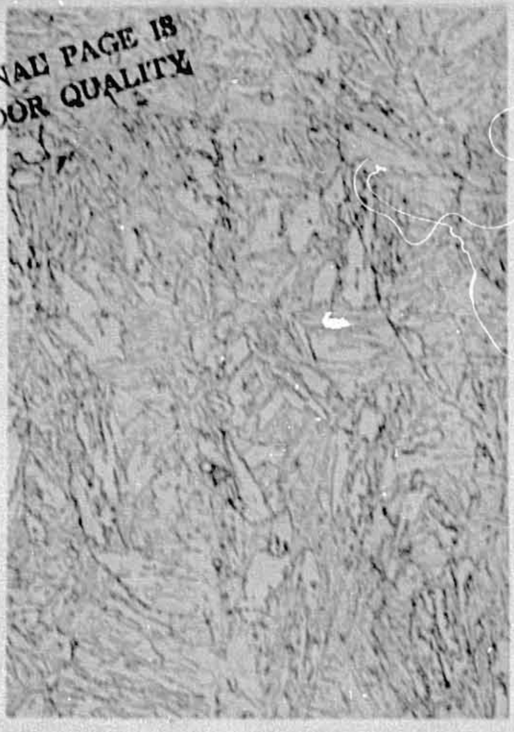


(b) VIM + ESR



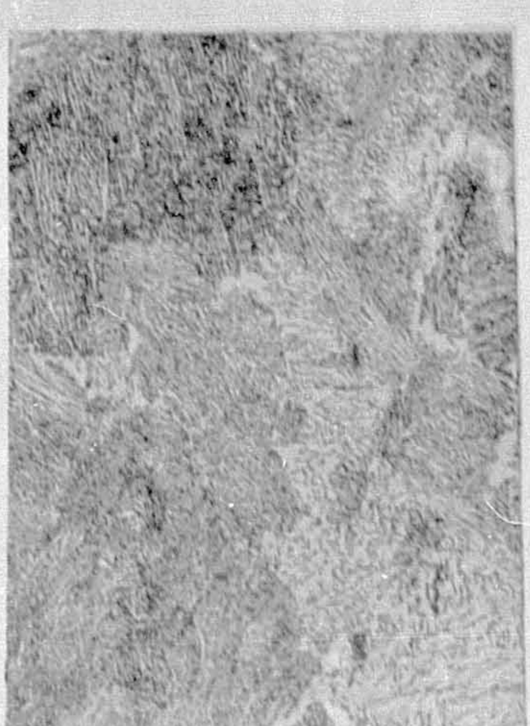
(c) VIM + ESR

FIGURE 6 . Typical inclusions in VIM, VIM+ESR grades of Fe-12Ni-0.2Ti (a & b) and Fe-12Ni-0.5Al (c) alloys.
Magnification x100, as rolled condition.



(a) As rolled

(b) Tempered & quenched at 550°C.

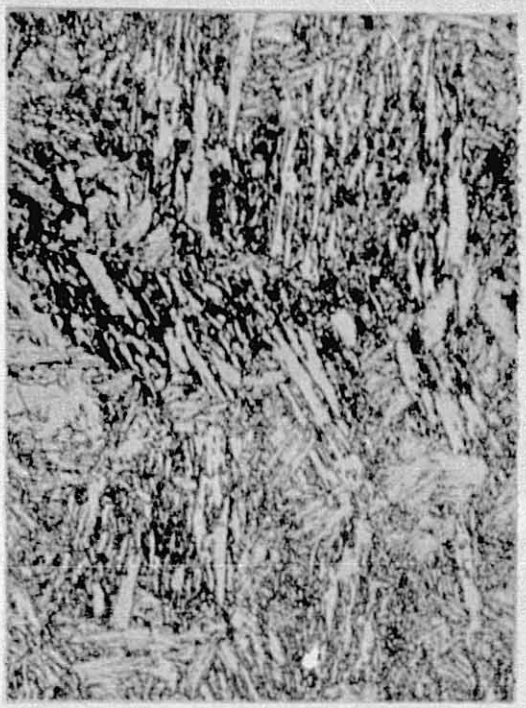


(c) Tempered & quenched at 685°C

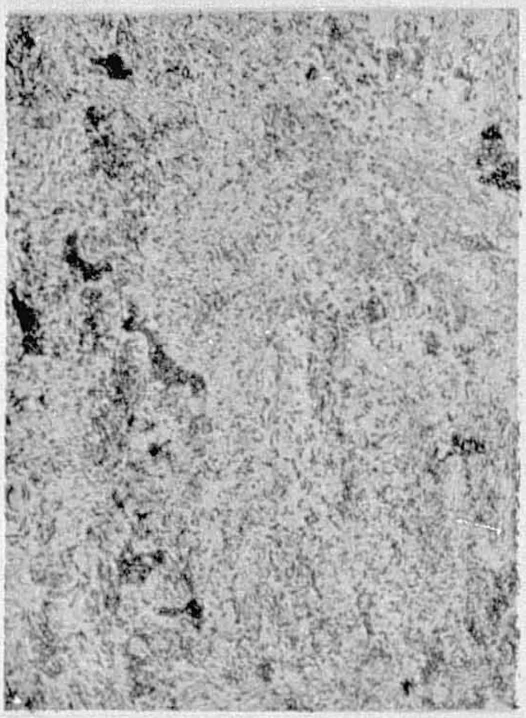
(d) Tempered & quenched at 820°C

FIGURE 7 . Microstructure of Fe-12Ni-2V alloy - AIM grade.
Magnification x1000.

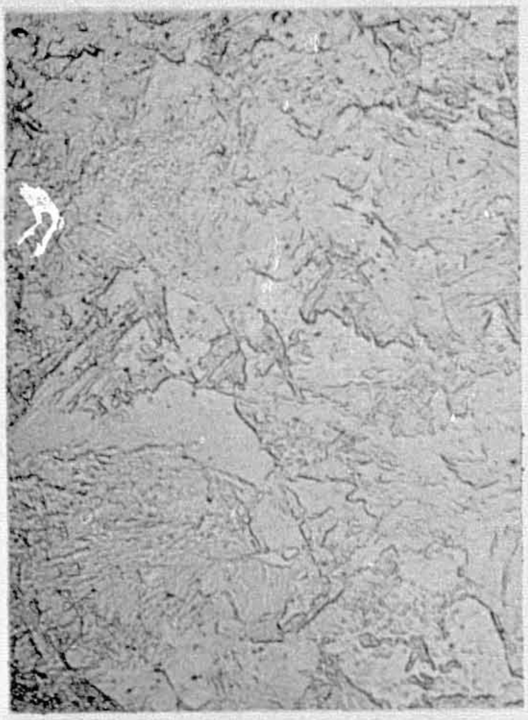
(a) As rolled



(b) Tempered & quenched at 550°C



(c) Tempered & quenched at 685°C

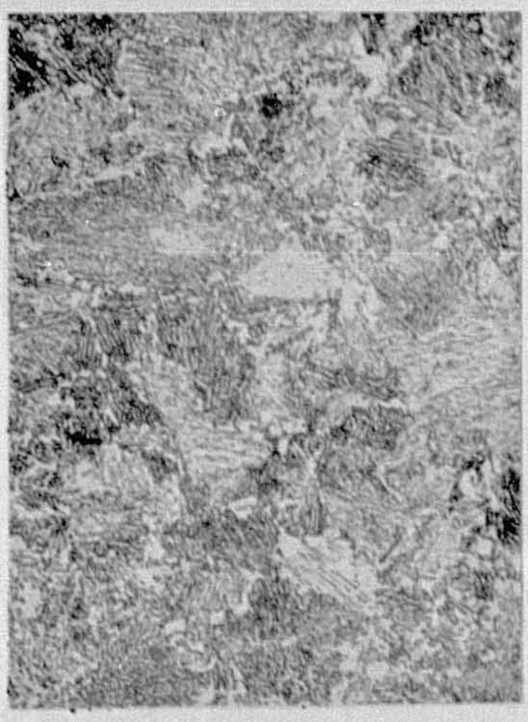


(d) Tempered & quenched at 820°C

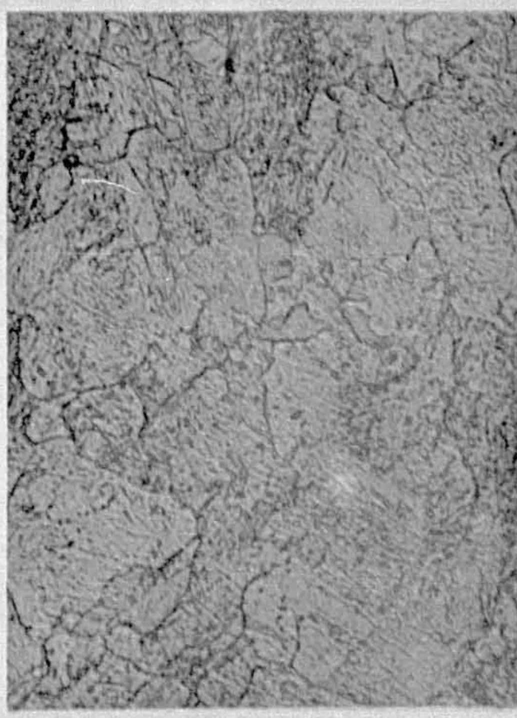
FIGURE 8 Microstructure of Fe-12Ni-2V alloy - AIM+ESR grades. Magnification x1000

(a) As rolled condition

(b) Tempered & quenched at 550°C



(c) Tempered & quenched at 685°C

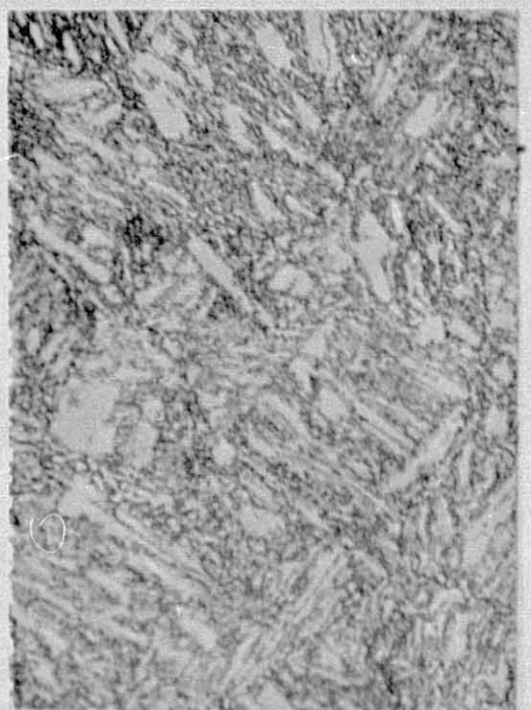


(d) Tempered & quenched at 820°C

FIGURE 9 . Microstructure of Fe-12Ni-2V alloy - VIM grade. Magnification x1000

(a) As rolled condition

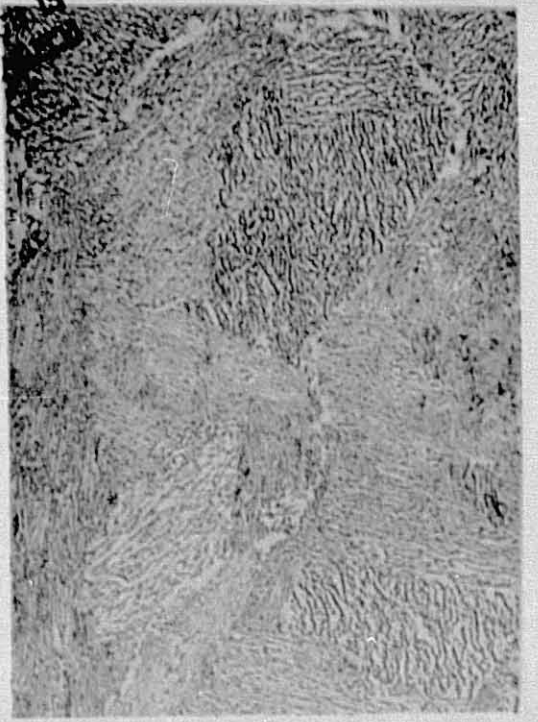
(b) Tempered & quenched at 550°C



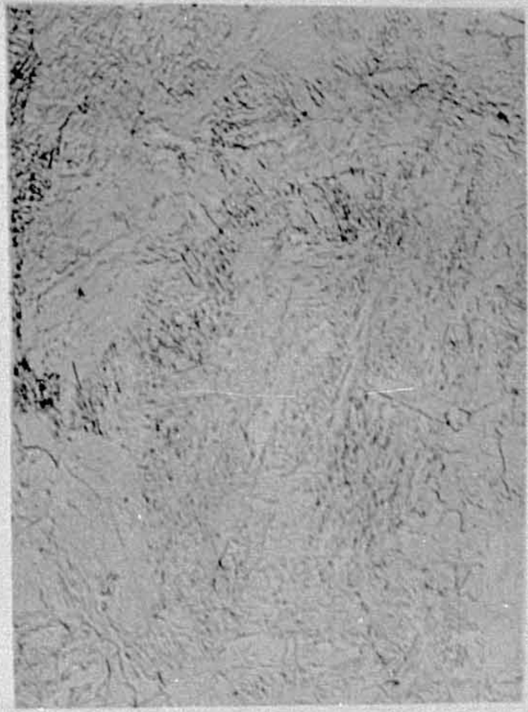
(c) Tempered & quenched at 600°C

(d) Tempered & quenched at 650°C

FIGURE 10 . Microstructure of Fe-12Ni-2V alloy - VIM+ESR grade. Magnification x1000

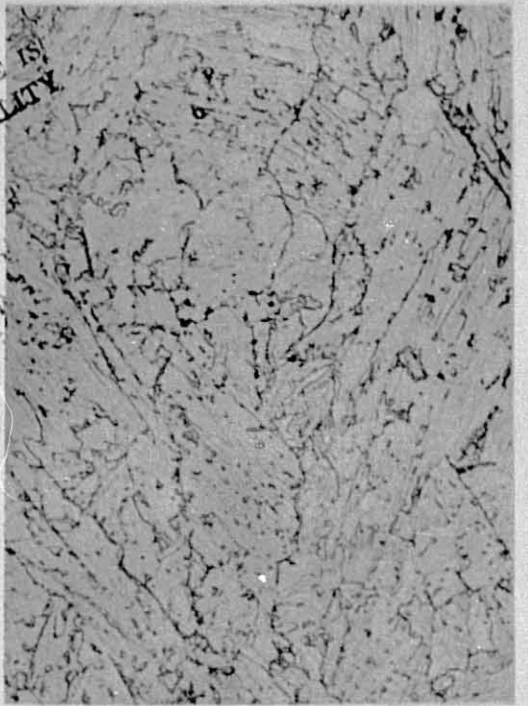


(e) Tempered & quenched at 685°C



(f) Tempered & quenched at 820°C

FIGURE 10 (continued). Microstructure of Fe-12Ni-2V alloy - VIM+ESR grade. Magnification x1000.



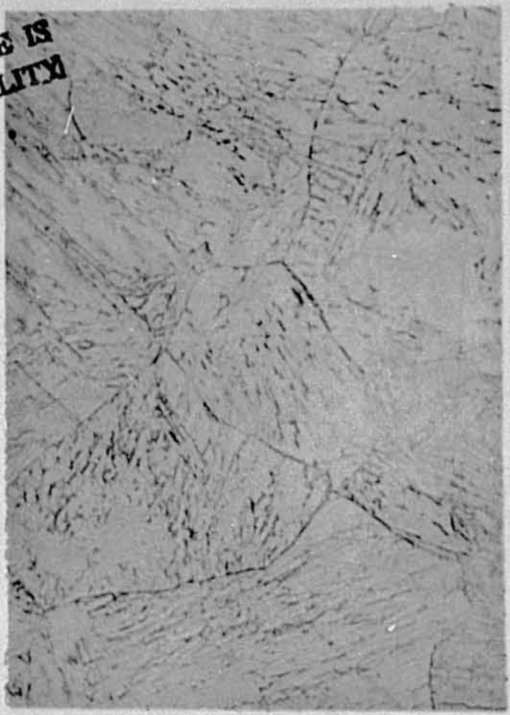
(a) As rolled condition

(b) Tempered & quenched at 550°C

(c) Tempered & quenched at 685°C

(d) Tempered & quenched at 820°C

FIGURE 11 . Microstructure of Fe-12Ni-0.2Ti alloy - VIM grades.
Magnification - x1000



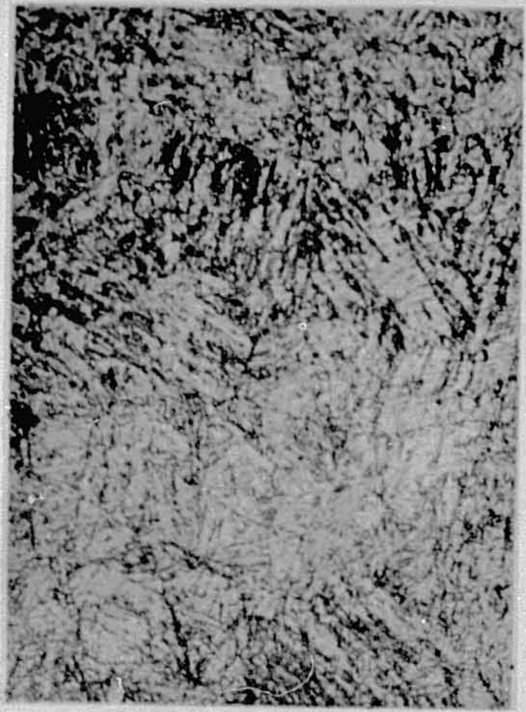
(a) As rolled condition

(b) Tempered & quenched at 550°C

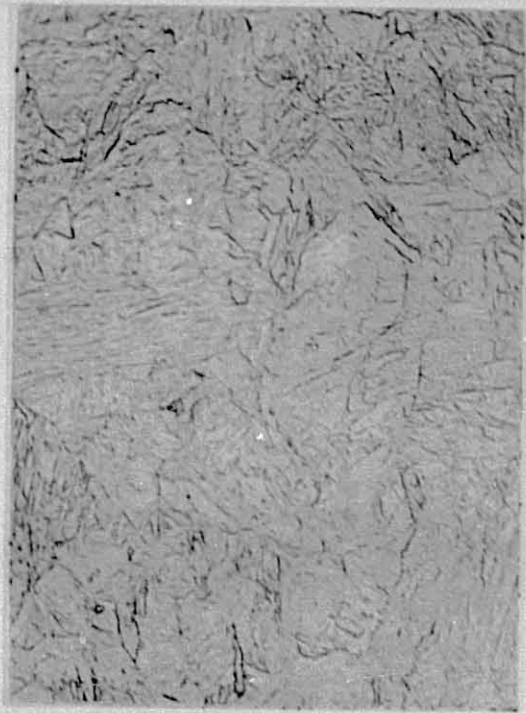
(c) Tempered & quenched at 600°C

(d) Tempered & quenched at 650°C

FIGURE 12 . Microstructure of Fe-12Ni-0.2Ti alloy - VIM+ESR grade. Magnification - x1000.

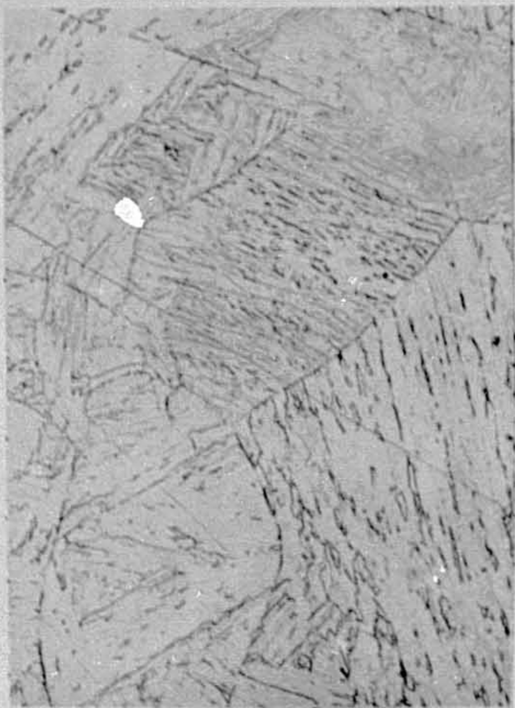


(e) Tempered & quenched at 685°C



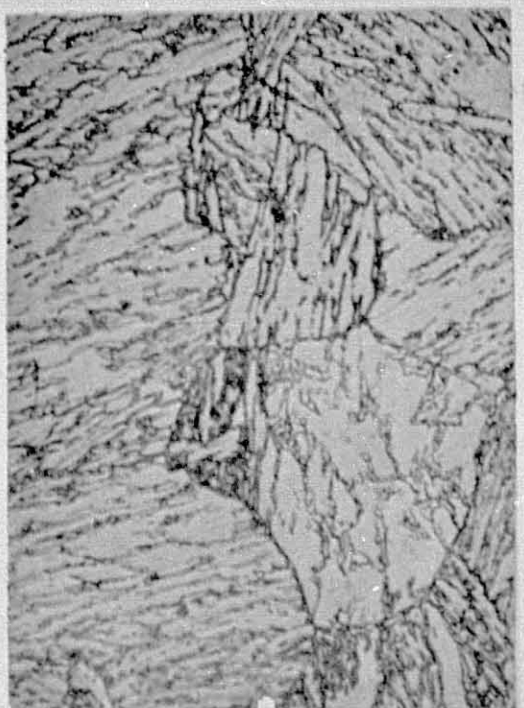
(f) Tempered & quenched at 820°C

FIGURE 12 (continued). Microstructure of Fe-12Ni-0.2Ti alloy - VIM+ESR grade. Magnification x1000.

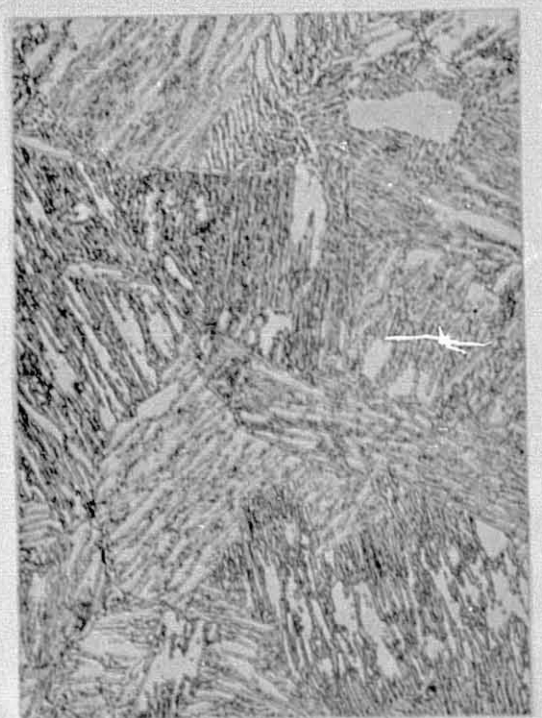


(a) As rolled condition

(b) Tempered & quenched at 500°C

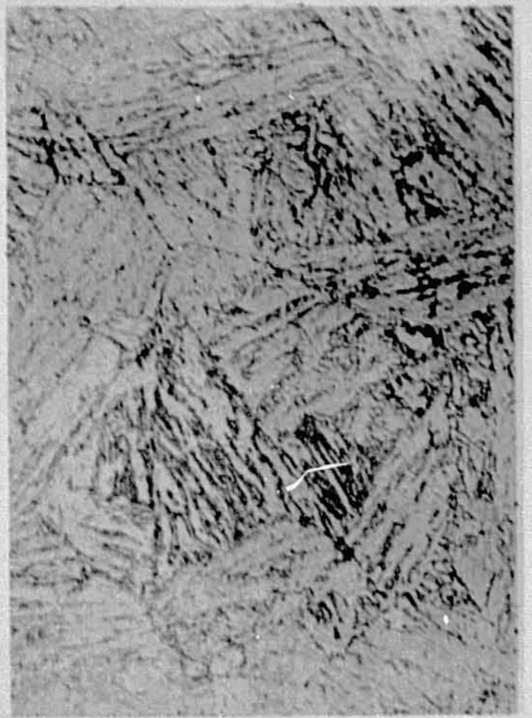


(c) Tempered & quenched at 550°C



(d) Tempered & quenched at 600°C.

FIGURE 13 . Microstructure of Fe-12Ni-0.5Al alloy - VIM+ESR grade. Magnification -x1000.



(e) Tempered & quenched at 685°C

(f) Tempered & quenched at 820°C

FIGURE 13 (continued). Microstructure of Fe-12Ni-0.5Al alloy - VIM+ESR grade. Magnification x1000.

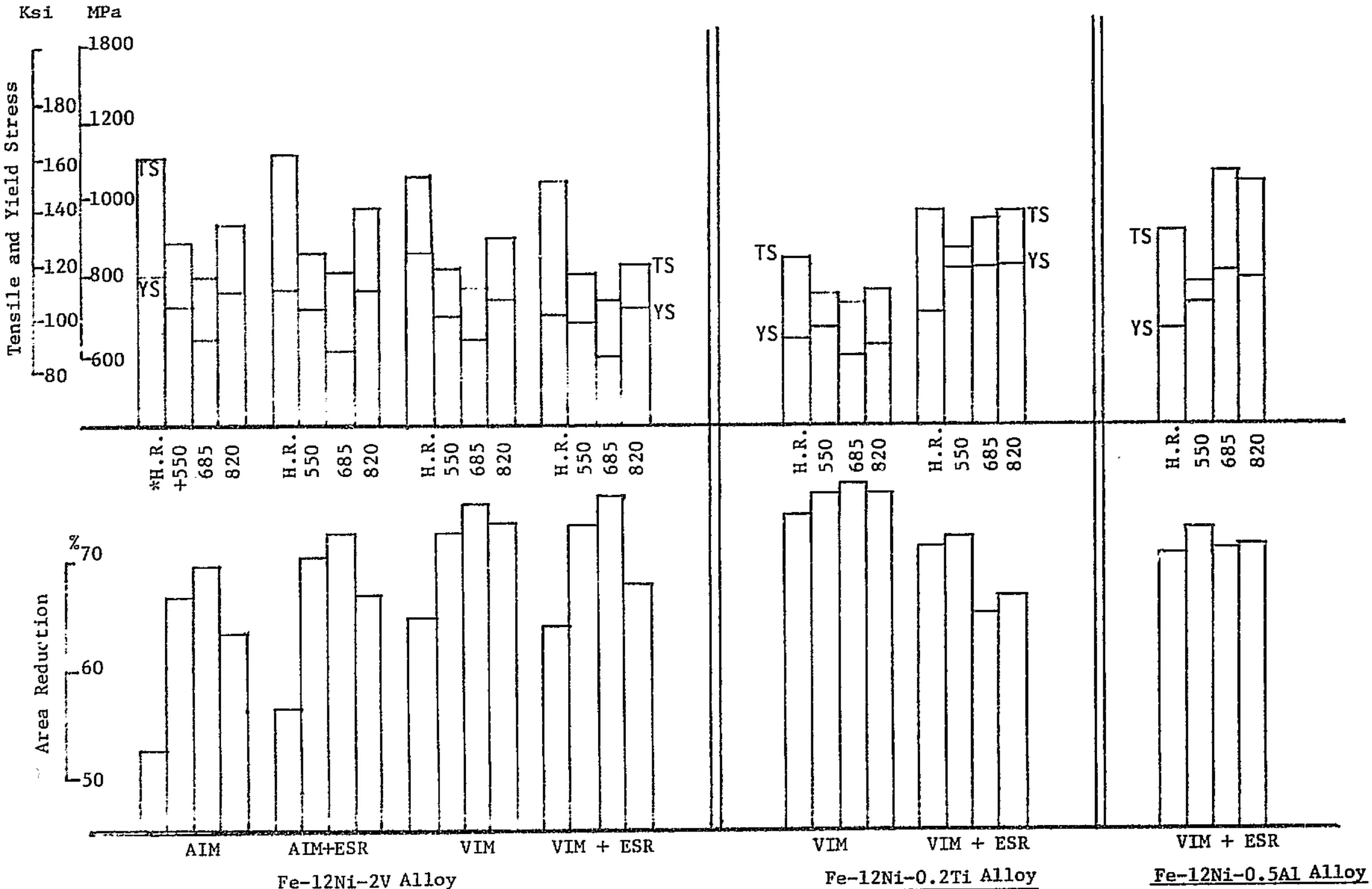


Figure 14 . Tensile properties of Fe-12Ni alloys as affected by different melting procedures and heat treatments for tests conducted at 25°C.

* H.R. = Hot Rolled Condition
\+ Tempering Temperature °C

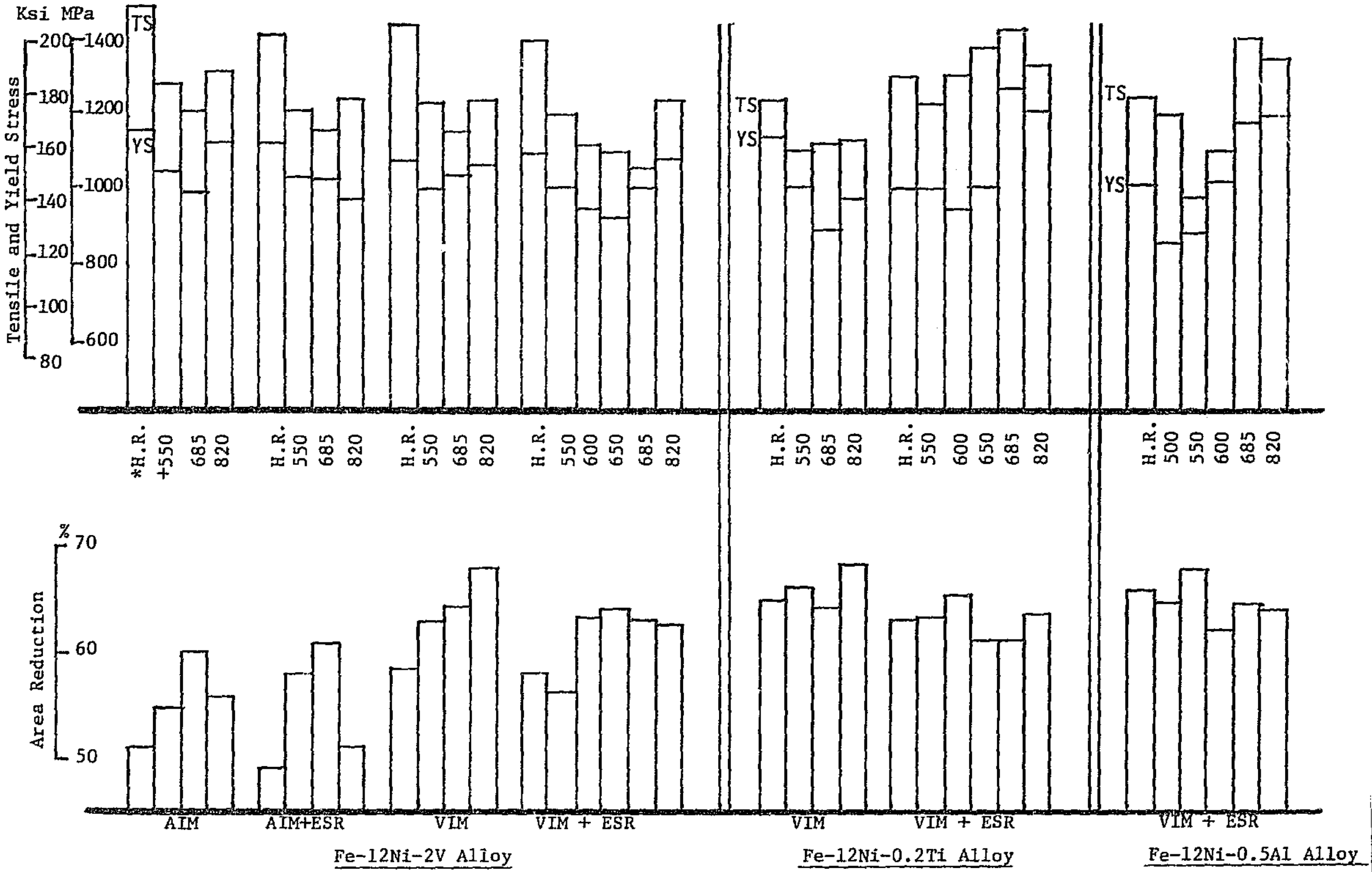


Figure 15. Tensile properties of Fe-12Ni Alloys as affected by different melting procedures and heat treatments for tests conducted at −196°C.

* H.R. = Hot Rolled Condition
\+ Tempering Temperature °C

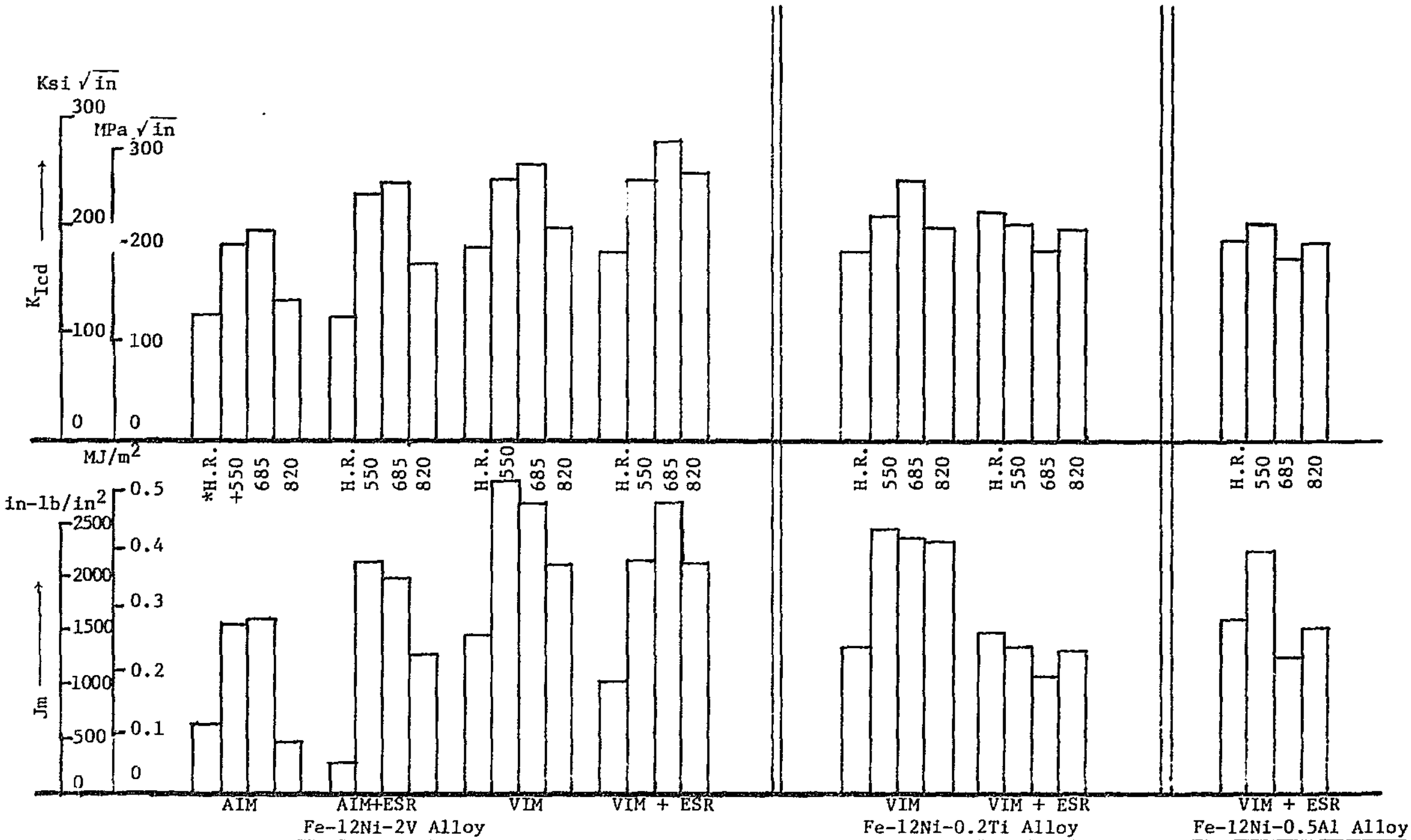


Figure 16 . Fracture toughness of Fe-12Ni alloys as affected by different melting and heat treatments for tests conducted at 25°C.

* H.R. = Hot Rolled Condition

\+ Tempering Temperature °C

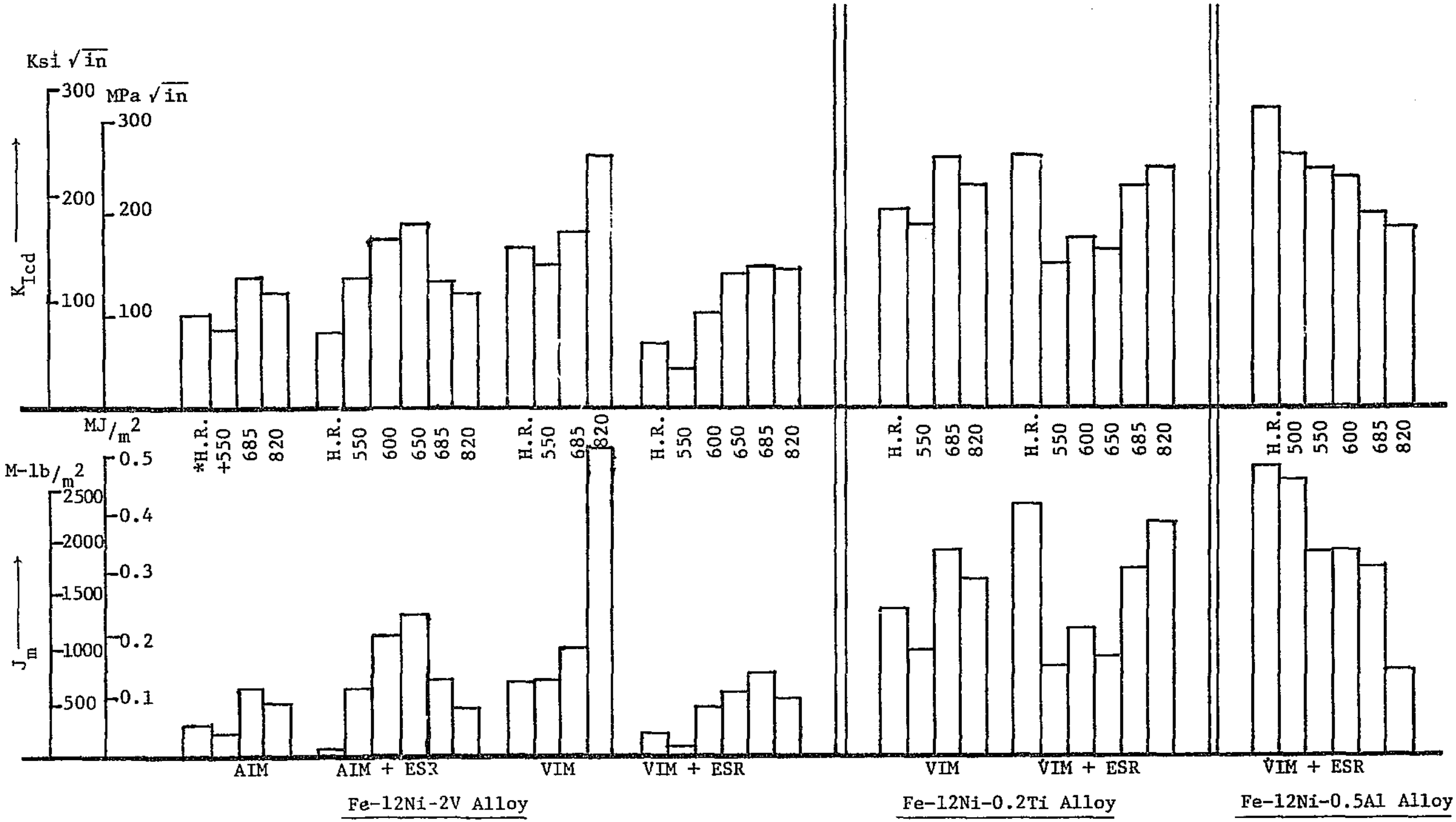


Figure 17 . Fracture toughness of Fe-12Ni alloys as affected by different melting procedures and heat treatments for tests conducted at -196°C.

* H.R. = Hot Rolled Condition
\+ Tempering Temperature °C