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Commercialization of Nickel and Iron Aluminides

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## Abstract

Metallurgists are taught during their college education that intermetallics are brittle phases and should be avoided in alloys of commercial interest. This education is so deeply rooted that irrespective of very significant advances made in the ductilization of aluminides, the road to their acceptance and commercialization is extremely tough. This paper identifies the requirements for commercialization of any new alloy and reports the activities carried out to commercialize nickel and iron aluminides. The paper also identifies areas which meet the current commercialization requirements and areas needing additional effort.

THE COMMERCIALIZATION OF ANY NEW STRUCTURAL MATERIAL typically takes 10 to 15 years. The 10- to 15-year range is typical for materials which are based on the conventional alloying concepts such as steels, solid-solution-strengthened alloys, and precipitation- and  $\gamma'$ -strengthened alloys. However, when one deals with the commercialization of intermetallics (aluminides), which have stereotyped as being very brittle materials, the commercialization times become even longer. We have made significant progress towards commercialization of nickel and iron aluminides, and this paper presents a comprehensive chronology of steps that were taken towards this commercialization effort.

## Background

The interest in developing intermetallic-based aluminides results from several scientific and engineering properties (1-13). Among the scientific aspects, intermetallics have ordered crystal structures which show low diffusion rates and resist dislocation movement. Among the engineering properties, nickel aluminides are of interest because of their high-temperature strength,

oxidation resistance, carburization resistance, and resistance in a chlorine environment. The iron-aluminide interest stems from its resistance to oxidizing and sulfidizing environments.

The above-stated advantages of ordered intermetallic aluminides have inspired scientists over the last four to five decades to develop the nickel and iron aluminides. Between the two, the  $Fe_3Al$ -based aluminides have undergone significantly more scientific investigations, probably because of their lower cost. However, in spite of numerous studies reported in the literature, only the recent aluminide compositions developed at the Oak Ridge National Laboratory (ORNL) have begun to receive commercial interest.

## Composition Development

The nickel-aluminide compositions are developed based on  $Ni_3Al$  with controlled additions of elements such as boron (14,15), zirconium, chromium (16,17), and molybdenum. The nickel-aluminide compositions developed with controlled additions are listed in Table I and includes the compositions of several commercial alloys that compete with or are being replaced by nickel aluminide. The iron-aluminide compositions (18-22) are based on  $Fe_3Al$  and contain controlled additions of boron, chromium, niobium, carbon, and zirconium. The  $Fe_3Al$  alloy compositions and the competitive alloys are summarized in Table II.

## Melting Practice

Even after the alloy compositions were identified, their melting posed a big resistance from commercial melters because of their high aluminum content. Typical concerns were the large difference in melting points of aluminum and nickel for nickel aluminides and aluminum and iron for iron aluminides, and the likelihood of preferential oxidation of aluminum. The commercial

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Table I. Chemical compositions of Ni<sub>3</sub>Al-based alloys and commercially available competing alloys

Element	Alloy, weight percent								
	IC-50 <sup>a</sup>	IC-396M <sup>b</sup>	IC-221M <sup>c</sup>	Haynes 214 <sup>d</sup>	HU <sup>e</sup>	Alloy 800 <sup>d</sup>	MORE 1	MORE 2	SUPERTHERM <sup>f</sup>
Al	11.3	7.98	8.0	4.5	-	0.4	-	0.75-1.50	-
Cr	-	7.72	7.7	16.0	18.0	21.0	25.0-28.0	32.0-34.0	26.0
Mo	-	3.02	1.43	-	-	-	-	-	-
Zr	0.6	0.85	1.7	-	-	-	-	-	-
B	0.02	0.005	0.008	-	-	-	-	-	-
C	-	-	-	0.03	0.55	0.05	0.40-0.50	0.15-0.25	0.5
Fe	-	-	-	3	42.45	45.5	-	-	16.2
Ti	-	-	-	-	-	0.4	-	-	-
Ni	88.08	80.42	81.1	76.35	39.0	32.5	35.0-38.0	48.0-52.0	35.0
Si	-	-	-	0.1	-	-	-	-	1.6
Y	-	-	-	0.02	-	-	-	-	-
W	-	-	-	-	-	-	1.25-2.00	15.0-17.0	5.0
Co	-	-	-	-	-	-	-	-	15.0
Mn	-	-	-	-	-	-	-	-	0.70

<sup>a</sup>Cold workable.

<sup>b</sup>Castable alloy for static applications (some microporosity).

<sup>c</sup>Castable alloy for dynamic application (minimum microporosity).

<sup>d</sup>Wrought alloy.

<sup>e</sup>Cast alloy.

<sup>f</sup>SUPERTHERM is a registered trademark of Abex Corporation.

Table II. Chemical compositions of Fe<sub>3</sub>Al-based alloys and competitive steel alloys

Element	Alloy, weight percent				
	FAS	FAL	FA-129	Type 422	Type 310 <sup>a</sup>
C	-	-	0.05	0.25 <sup>b</sup>	0.08 <sup>b</sup>
Cr	2.2	5.5	5.5	12.0	25.0
Al	15.9	15.9	15.9	-	-
B	0.01	0.01	-	-	-
Zr	-	0.15	-	-	-
Nb	-	-	1.0	-	-
Mn	-	-	-	-	2.0 <sup>b</sup>
Si	-	-	-	-	1.50 <sup>b</sup>
Mo	-	-	-	1.0	-
V	-	-	-	0.25	-
W	-	-	-	1.0	-
Ni	-	-	-	0.8	20.0
Fe	c	c	c	c	c

<sup>a</sup>Wrought composition.

<sup>b</sup>Maximum.

<sup>c</sup>Balance.

melting concerns were eliminated by the development of the Exo-Melt™ process (23-24). This process is based on the fact that the formation of aluminides from their constituent elements is exothermic (see Table III) and utilizes a furnace-loading sequence to effectively use the heat of formation in the melting process. A typical furnace-loading scheme for melting Ni<sub>3</sub>Al-based alloy IC-221M is shown in Figure 1. The Exo-Melt™ process not only eliminates the melting-point difference issue but also provides a safe and economical commercial melting process with the use of currently available melt technology at most foundries. Several advantages of the Exo-Melt™ process are summarized in Table IV.

Table III. Heats of formation data for nickel and iron aluminides

Intermetallic	Heat of formation $\Delta H_f^{298}$ (K cal/mole)	Weight percent aluminum	Melting point (°C)
Ni <sub>3</sub> Al	-36.6 ± 1.2	13.28	1395
NiAl	-28.3 ± 1.2	31.49	1639
Ni <sub>2</sub> Al <sub>3</sub>	-67.5 ± 4.0	40.81	1133
NiAl <sub>3</sub>	-36.0 ± 2.0	57.96	854
Fe <sub>3</sub> Al	-16.0	13.87	1502
FeAl	-12.0	32.57	1215
FeAl <sub>2</sub>	-18.9	49.10	1164
Fe <sub>2</sub> Al <sub>5</sub>	-34.3	54.70	1171

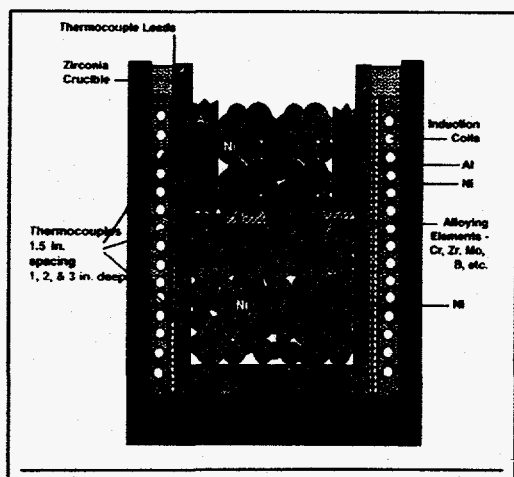


Figure 1 - A schematic of the furnace-loading sequence employed for the Exo-Melt™ process to melt nickel aluminides. A similar arrangement can be used to melt iron aluminides.

Table IV. Benefits of Exo-Melt™ process over conventional process for melting aluminides

Feature	Melting process	
	Conventional	Exo-Melt™
Power use	Unit power	One-half the power of the conventional method
Time to melt	Unit time	One-half the melting time of the conventional method
Cost	Unit cost	One-half the cost of the conventional method
Safety	Safety issue due to excessive heat	No safety issue because of controlled process
	Safety issue due to excessive wear of the crucible	No overheating of crucible
Melt temperature	No control	Real control
Crucible life	Limited due to overheating and excessive wear	Extended life due to temperature control
Melt composition	No control	Real control
Oxide inclusions	Excessive inclusions due to overheating	Very low due to good temperature controls and short melt times
Industrial acceptance	Not many companies interested due to safety concerns	Used successfully by several companies

### Compositional Control

Alloy design has shown that control of Ni/Al or Fe/Al ratios is critical for obtaining ductility and toughness in aluminides. Such control of chemistry requires knowledge of the recovery of various elements and, more importantly, the methods of analyzing them accurately. Regarding the recovery of elements, our experience with the melting of aluminides is described here.

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**Ni<sub>3</sub>Al-Based IC-221M Alloy.** For a virgin melt, using the furnace-loading sequence shown in Figure 1, the only element that recovers less than 100% is zirconium. This is the only element that needs to be charged one to two times the desired value of 1.7 wt % and must be analyzed prior to furnace tapping. There are no issues for boron recovery; therefore, it should not be overcharged.

The other elements, although not specified but needing control, are carbon and silicon. The carbon can be picked up from the melt stock; therefore, care should be taken to keep its value less than 0.05 wt %. The control of silicon content is important because aluminum in the aluminides can reduce SiO<sub>2</sub> from the furnace lining or from the reaction of molten aluminide with the sand that is often used for pigging the unused melt stock. For weldable structures, the silicon content should be kept below 0.05 wt %, which can be achieved by minimizing the holding time of the melt stock in the furnace and ladles or by cleaning off any sand adhered to the surface of the castings. Certain melt treatment may also be possible to remove the silicon from the melt prior to pouring. No work is in progress at ORNL or at commercial producers' facilities in this area.

**Fe<sub>3</sub>Al-Based Alloys.** Table V presents the recovery data on many heats of Fe<sub>3</sub>Al which have been developed. As noted in this table, the recovery of chromium, aluminum, and boron is less than 100%, and proper adjustment is required to obtain the final composition. To ensure porosity-free material, iron aluminide melting requires special care in using dry charge.

The on-line chemical analysis capability is important to ensure the proper final chemistry. There are no primary standards currently available for either nickel or iron aluminides. Using the secondary standards

produced at ORNL, at least two casting houses have on-line chemical analysis capability for nickel aluminides. No such capability yet exists for iron aluminides. However, when the market for iron aluminides demands such standards, ORNL will work with potential producers in a manner similar to that used for the Ni<sub>3</sub>Al-based alloys.

## Processing

The ease of processing aluminides into various product forms, using conventional processing equipment, determines the favorable processing route and their usefulness for applications. The current processing status of each of the Ni<sub>3</sub>Al- and Fe<sub>3</sub>Al-based aluminides is described below.

**Ni<sub>3</sub>Al-Based Alloys.** Sand and centrifugal castings are currently the best methods for low-cost production of nickel-aluminide components, and the best casting alloy is IC-221M. The IC-50 can also be sand- and centrifugal-cast into shapes, however, it is more susceptible to casting shrinkage. It can also be cold-processed into sheet or wire with intermediate anneals at 1100°C.

The IC-218LZr is the only nickel-aluminide composition that can be hot-processed (25) from ingots into bars or plate. However, the hot-processing requires good control of temperature, which can be accomplished by canning the alloy in a carbon steel can. Hot-processed IC-218LZr can be cold-processed in a manner similar to IC-50.

**Fe<sub>3</sub>Al-Based Alloys.** The Fe<sub>3</sub>Al-based alloy ingots can be processed (26,27) best by hot-working operations. The hot-working temperatures are typically in the range of 1000 to 1200°C. The hot-worked bars and plates can

Table V. Recovery of various elements during air-induction melting of 7-kg iron-aluminide heats

Element	FAS		FAL		FA-129	
	Target (wt %)	Recovery (%)	Target (wt %)	Recovery (%)	Target (wt %)	Recovery (%)
C	--	--	--	--	0.04	250.00 <sup>a</sup>
Cr	2.194	95.72	5.46	91.03	5.46	95.24
Mo	--	--	--	--	--	--
Nb	--	--	--	--	0.21	100.00
Al	15.932	96.67	15.88	96.73	15.88	97.54
B	0.011	27.27	0.01	80.00	--	--
Zr	--	--	0.19	100.00	--	--
Fe	<i>b</i>	--	<i>b</i>	--	<i>b</i>	--

<sup>a</sup>Recovery of greater than 100% indicates pickup of carbon from external sources. For example, a graphite rod was used for stirring the liquid metal, and a small fraction of the graphite was probably dissolved in the metal.

<sup>b</sup>Balance (100 minus total of all other elements).

only be warm-processed between 650 to 800°C. The Fe<sub>3</sub>Al-based alloys can also be sand-cast into shapes. However, no significant applications of cast product have yet been identified.

The water-atomized powders of Fe<sub>3</sub>Al-based alloys can be sintered into product of controlled porosity. Such products are used as candle filters for hot-gas clean-up.

## Properties

Knowing the physical and mechanical properties of aluminides is essential for their broad acceptance for various applications. The current status of properties is presented below.

**Physical Properties.** The density and melting-point data for nickel and iron aluminides and their competitive alloys are summarized in Table VI. The elastic modulus data for nickel and iron aluminides are presented in Tables VII and VIII. The thermal expansion, thermal conductivity, and heat-capacity data for nickel and iron aluminides are presented in Tables IX and X.

**Hardness.** Room-temperature hardness of nickel-aluminide alloy IC-221M and the competitive HU alloy in the as-cast and after a 1-h treatment at various temperatures are plotted in Figure 2. This figure shows that IC-221M is thermally stable to 1-h treatments for temperatures up to 1150°C. However, the HU alloy undergoes a reaction with treatment at 800°C. The peak hardness occurs after a 800°C treatment and then drops because of precipitate coarsening with higher treatment temperature.

The hot-hardness data on cast nickel-aluminide alloys (IC-50 and IC-221M) are compared with the data on the HU alloy in Figure 3. Data in this figure show that nickel aluminide is significantly stronger than HU for the entire range of test temperatures. The hot-hardness data for a Fe<sub>3</sub>Al alloy, FAL, in the wrought condition and a FeAl alloy, FA-386M1, (see Table II for composition) in the cast condition are compared with the data for cast nickel-aluminide alloys in Figure 4. Data in this figure show that the Fe<sub>3</sub>Al alloy is significantly weaker than the Ni<sub>3</sub>Al-based alloy, especially at temperatures above 600°C. The FeAl alloy shows strength between Fe<sub>3</sub>Al and Ni<sub>3</sub>Al alloys.

**Tensile Properties.** The tensile properties (0.2% yield strength, ultimate tensile strength, and total elongation) for a large number of cast test bars of alloy IC-221M are plotted in Figure 5. The test bars were either sand-cast, investment-cast, or electrodischarge-machined (EDM) from cast ingots. The test bars also represent over 15 different heats and a pouring temperature range over 100°C. The average properties from data in Figure 5 are compared with the data on competitive alloys in Figure 6. This figure shows that the cast nickel-aluminide alloy IC-221M is significantly stronger than the HU and SUPERTHERM™ alloys up to 1100°C. At 1175°C and higher, the IC-221M alloy falls in strength somewhat below that of the SUPERTHERM™.

The average tensile properties of Fe<sub>3</sub>Al-based alloys FAS, FAL, and FA-129 in the wrought condition are compared with data for a ferritic stainless steel (type 422) and austenitic stainless steel (type 310) in Figure 7. This figure shows that the heat-treatable ferritic steel type 422 has significantly higher yield strength, somewhat higher ultimate tensile strength, and lower elongation than the three Fe<sub>3</sub>Al-based alloys of our study. The austenitic stainless steel type 310 sheet shows lower yield strength up to 700°C, lower ultimate tensile strength up to 600°C, and significantly higher ductility than the Fe<sub>3</sub>Al-based alloys.

**Creep Properties.** The creep data for cast IC-221M are compared with several competitive alloys in Figure 8. For the test conditions where data are available, the IC-221M alloy is stronger than all of the competitive alloys. However, when data are linearly extrapolated, the strength difference between IC-221M and SUPERTHERM™ becomes essentially negligible. Creep tests at higher temperature and longer duration are currently under way to confirm the validity of linear extrapolation. The creep data for the Fe<sub>3</sub>Al-based alloys in the wrought condition are compared with types 422 and 310 stainless steels in Figure 9. This figure shows that the Fe<sub>3</sub>Al-based alloys are weaker in creep than the ferritic type 422 and austenitic type 310 stainless steels. ORNL's efforts to enhance the creep strength of Fe<sub>3</sub>Al-based alloys are described in a paper by Stoloff and McKamey (28).

**Thermal Aging.** The effect of thermal aging on properties of cast nickel aluminide are investigated by testing samples exposed in the laboratory furnaces and those removed from production furnaces. Exposure temperatures for the laboratory furnace were 850, 1050, and 1100°C. The samples removed from service were exposed at approximately 900°C for two years and approximately 1250°C for one year. The laboratory-exposed specimens also included the HU alloy. The change in room-temperature hardness of IC-221M and the HU alloy after 1050 and 1100°C exposures are plotted in Figure 10. These data show that room-temperature hardness of IC-221M is reduced by exposures at 1050 and 1100°C. It appears that after the initial drop, the hardness values stabilize for exposure at 1050°C. However, the exposure at 1100°C has not yet resulted in a similar stable hardness value. The HU alloy [see Figure 10(b)] has much lower hardness than IC-221M, but it shows only minor changes with exposures at 1050 and 1100°C.

The hot-hardness data on a coupon of IC-396M (very similar composition to IC-221M) removed after two years of exposure from a production furnace operating at approximately 900°C are plotted in Figure 11. This figure shows that cast nickel aluminide after 2-year exposure at approximately 900°C has only a small drop across the entire test-temperature range. Tensile tests on thermally aged specimens are currently under way.

**Corrosion Properties.** The Ni<sub>3</sub>Al-based alloys provide significant benefit in their resistance to oxidation,

Table VI. Density and melting point of Ni<sub>3</sub>Al- and Fe<sub>3</sub>Al-based alloys as compared to commercial alloys

Alloy	Density		Melting temperature	
	lb/cu-in.	g/cm <sup>3</sup>	°F	°C
<b>Nickel aluminides and competitive alloys</b>				
IC-221M	0.284	7.86	2140 <sup>a</sup> to 2467	1171 <sup>a</sup> to 1353
IC-218LZr	0.281	7.77	2529 <sup>b</sup>	1387
IC-50	0.275	7.60	2543	1395
Alloy 800	0.287	7.94	2475 to 2525	1357 to 1385
HU	0.290	8.02	2450	1343
SUPERTHERM™	0.297	8.22	—	—
<b>Iron aluminides and competitive alloys</b>				
FAS	0.236	6.53	2606 to 2691	1430 to 1477
FAL	0.235	6.51	2606 to 2691	1430 to 1477
FA-129	0.236	6.54	2606 to 2691	1430 to 1477
Type 422	0.280	7.78	2675 to 2700	1468 to 1482
Type 310	0.290	8.02	2550 to 2650	—

<sup>a</sup>Eutectic temperature.

<sup>b</sup>No eutectic formation.

Table VII. Elastic modulus of Ni<sub>3</sub>Al-based alloys and competitive alloys

Temperature (°C)	Young's Modulus (GPa)					
	Alloy					
	IC-221M <sup>a</sup>	IC-218LZr <sup>a</sup>	IC-50 <sup>a</sup>	Alloy 800	HU	SUPERTHERM™
20	200.0	225.0	203.5	196.5	186.2	178.5
150	195.0	223.0	198.0	188.0	—	<i>b</i>
300	184.0	218.0	—	178.3	—	<i>b</i>
500	163.0	206.5	178.0	171.6	—	<i>b</i>
700	157.0	193.5	162.5	150.1	—	133.8
900	139.0	—	136.0	<i>b</i>	—	112.4
1100	114.0	170.0	127.0	<i>b</i>	—	89.6

<sup>a</sup>Determined by Dynamic method.

<sup>b</sup>Data not available.

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Table VIII. Elastic modulus of Fe<sub>3</sub>Al-based alloys and competitive alloys

Temperature (°C)	Young's Modulus (GPa)				
	Alloy <sup>a</sup>				
	FAS	FAL	FA-129	Type 422 stainless steel	Type 310 stainless steel
23	183.0	201.0	207.0	207.0	200.0
150	178.0	193.0	198.0	200.0	190.0
300	167.0	179.0	<i>b</i>	190.0	176.0
500	159.5	160.5	166.0	155.1	162.0
700	135.0	148.0	144.0	-	141.4
900	116.5	134.5	130.0	-	110.3
1100	100.0	108.5	108.0	-	62.1

<sup>a</sup>All data on sheet material.<sup>b</sup>Not available.

Table IX. Thermal expansion and thermal conductivity of cast IC-221M and cast competitive alloys

Temperature (°C)	Alloy					
	IC-221M		HU		SUPERTHERM™	
	Mean coefficient of expansion (10 <sup>-6</sup> /°C) <sup>a</sup>	Thermal conductivity (w/m·k)	Mean coefficient of expansion (10 <sup>-6</sup> /°C) <sup>b</sup>	Thermal conductivity (w/m·k)	Mean coefficient of expansion (10 <sup>-6</sup> /°C)	Thermal conductivity (w/m·k)
100	12.77	11.9	17.28 <sup>b</sup>	4.04	18.36 <sup>c</sup>	4.10
200	13.08	13.9		4.62		4.73
300	13.40	15.2		5.08		5.26
400	13.72	16.7		5.54		5.78
500	14.01	18.0		5.95		6.35
600	14.33	20.3		6.58		6.93
700	14.72	23.0		7.16		7.51
800	15.17	25.2		7.62		8.09
900	15.78	27.5 <sup>d</sup>		8.32		8.66
1000	16.57	30.2 <sup>d</sup>		8.95		9.12

<sup>a</sup>Room temperature to specified temperature.<sup>b</sup>Room temperature to 982°C.<sup>c</sup>Room temperature to 1204°C.<sup>d</sup>Extrapolated from data up to 800°C.

Table X. Thermal expansion and thermal conductivity of wrought FA-129 alloy and competitive alloys

Temperature (°C)	Alloy					
	FA-129		Type 422 stainless steel		Type 310 stainless steel	
	Mean coefficient of expansion ( $10^{-6}/^{\circ}\text{C}$ ) <sup>a</sup>	Thermal conductivity (w/m-k)	Mean coefficient of expansion ( $10^{-6}/^{\circ}\text{C}$ ) <sup>a</sup>	Thermal conductivity (w/m-k)	Mean coefficient of expansion ( $10^{-6}/^{\circ}\text{C}$ ) <sup>a</sup>	Thermal conductivity (w/m-k)
100	15.41	b	11.16	8.26	14.76	4.76
200	15.88	b	11.16	8.55	15.66	5.66
300	16.70	b	11.34	8.89	16.20	6.06
400	17.93	b	11.43	9.18	16.47	6.64
500	19.18	b	11.70	9.30	16.74	7.51
600	20.37	b	12.06	9.33	16.83	8.37
700	21.00	b	-	-	18.00	9.53
800	21.84	b	-	-	18.36	10.05
900	22.79	b	-	-	18.90	10.80
1000	23.38	b	-	-	19.08	-
1100	23.37	b	-	-	-	-
1200	23.46	b	-	-	-	-

<sup>a</sup>Room temperature to specified temperature.

<sup>b</sup>Not available.

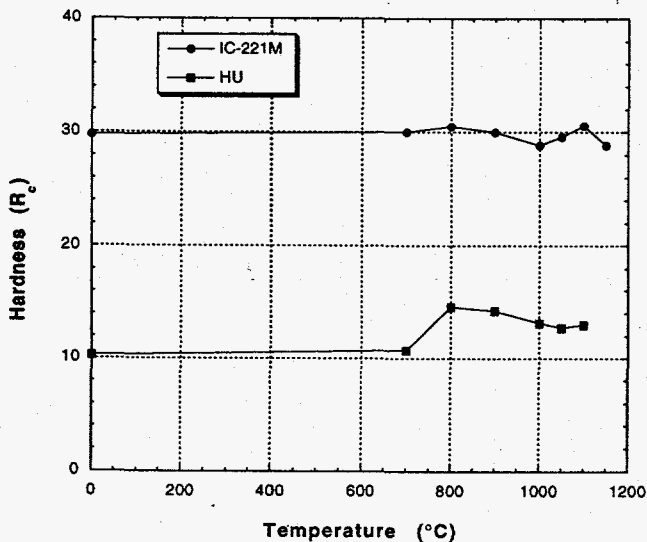


Figure 2 - Room-temperature hardness as a function of 1-h heat treatment at various temperatures for cast IC-221M and HU alloys.

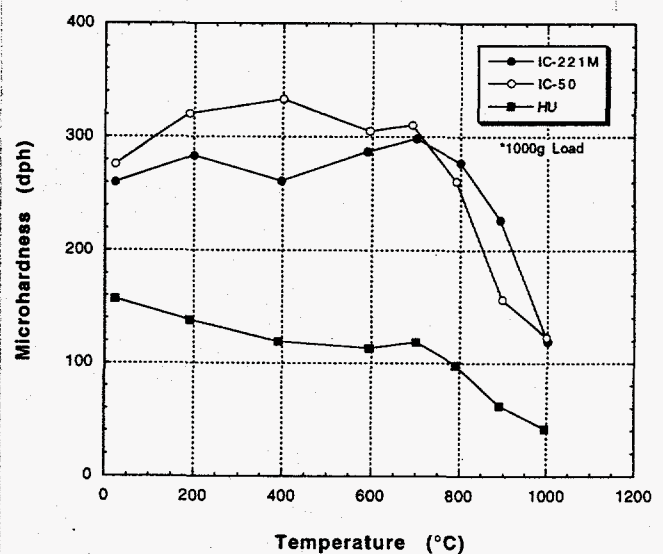


Figure 3 - Comparison of hardness as a function of test temperature for cast Ni<sub>3</sub>Al-based alloys.

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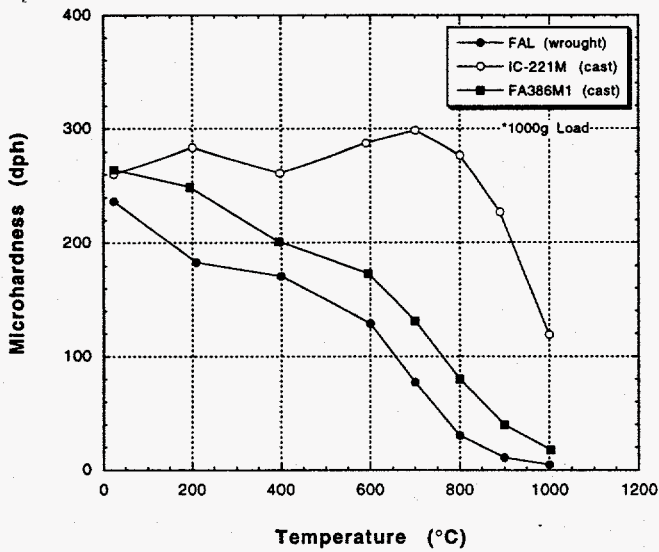


Figure 4 - Comparison of hardness as a function of test temperature for nickel- and iron-aluminide alloys.

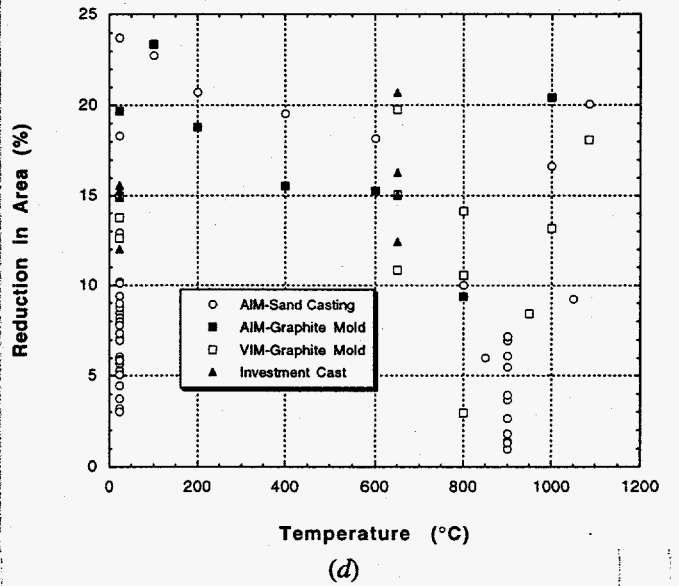
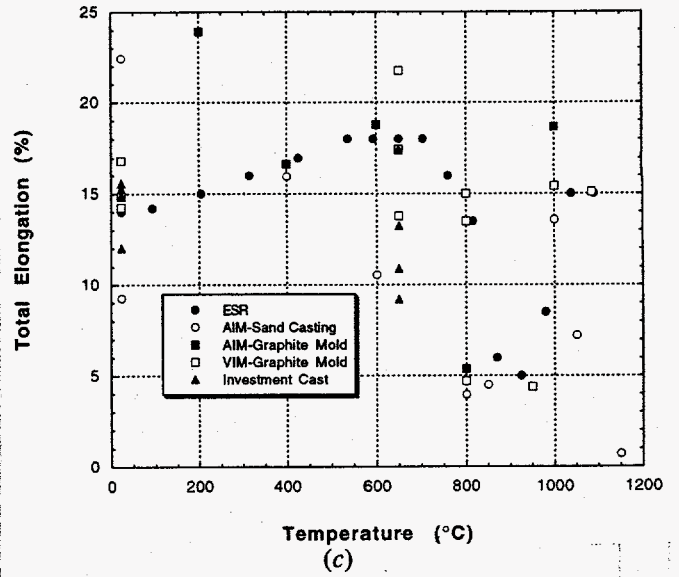
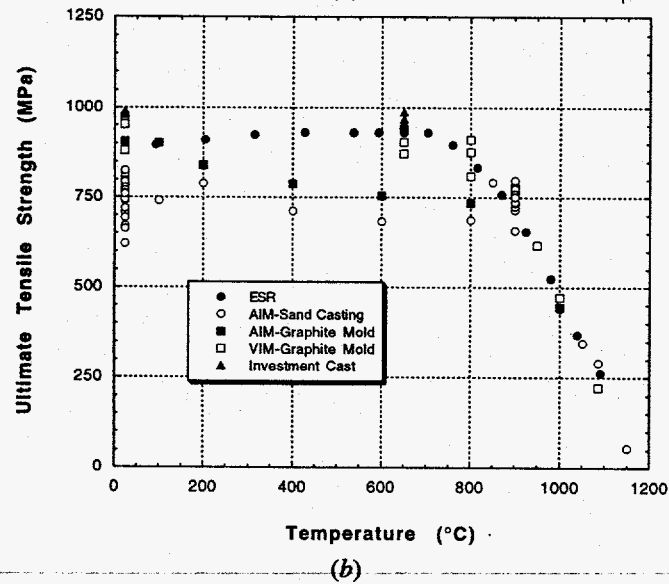
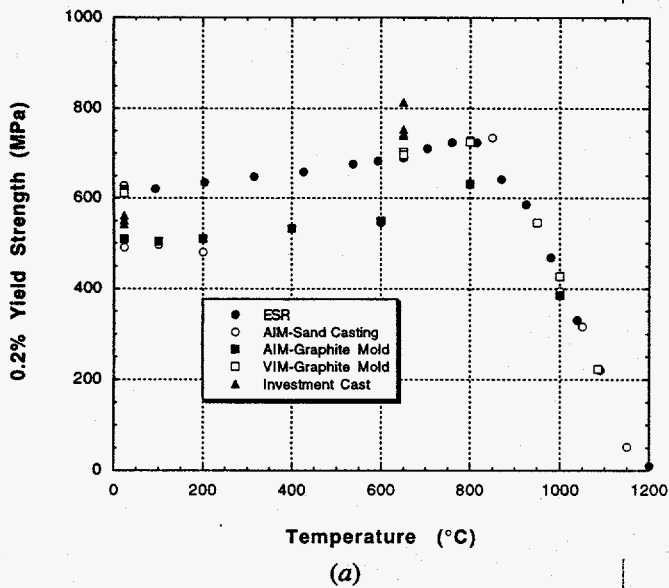
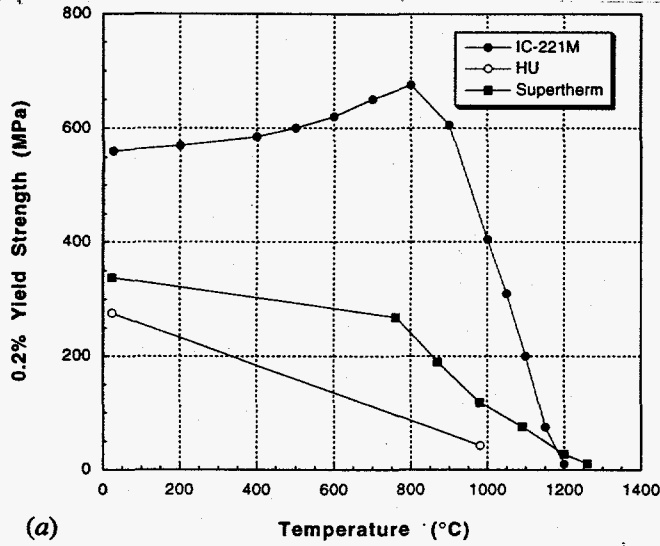


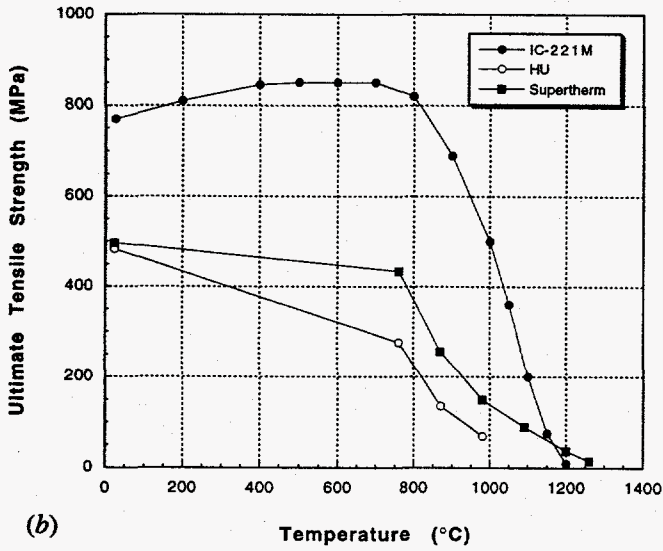
Figure 5 - Tensile properties of cast test bars of Ni<sub>3</sub>Al-based alloy IC-221M: (a) yield strength, (b) ultimate tensile strength, (c) total elongation, and (d) reduction of area.



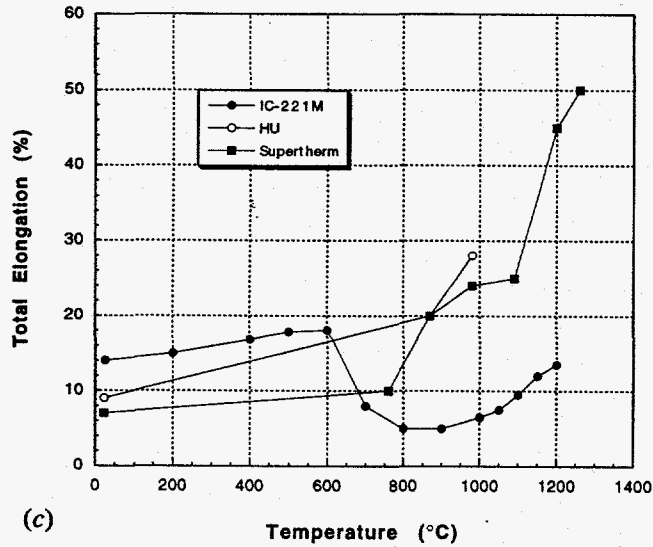
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(a)

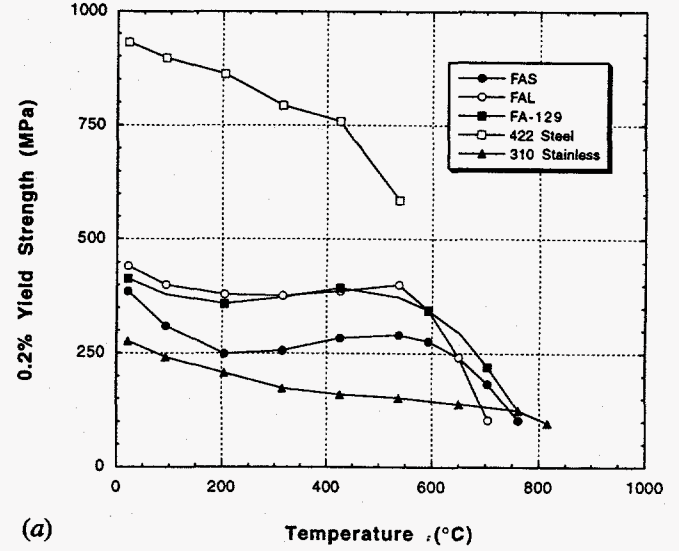


(b)

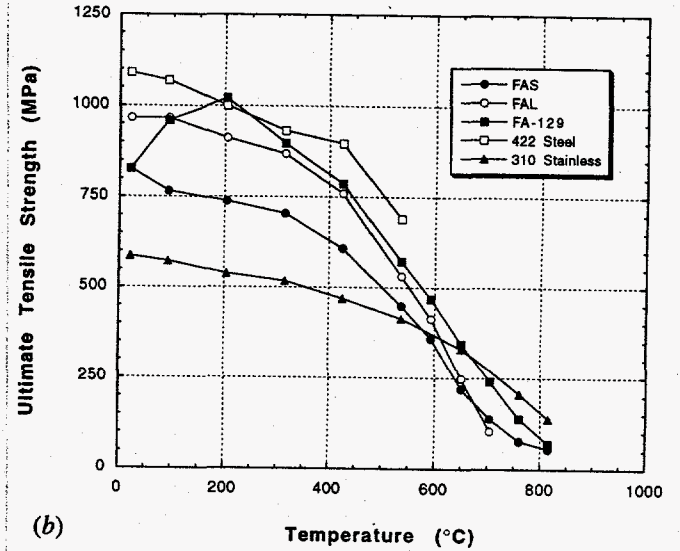


(c)

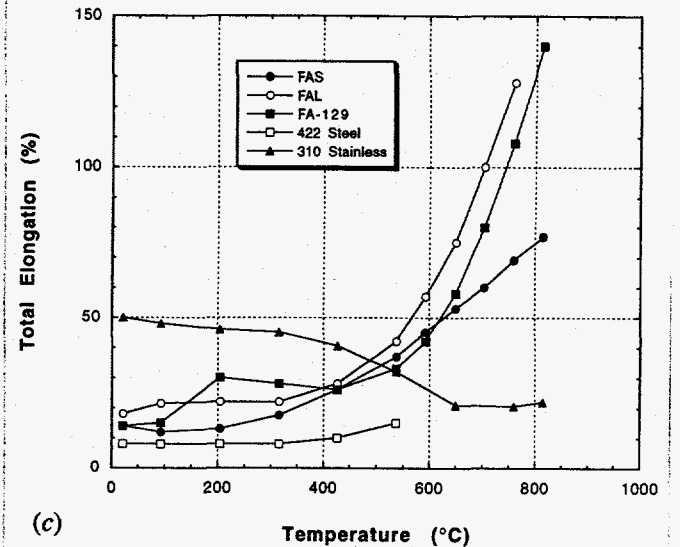
Figure 6 - Comparison of average tensile properties of cast IC-221M with that of cast HU and SUPER THERM™: (a) yield strength, (b) ultimate tensile strength, and (c) total elongation.



(a)

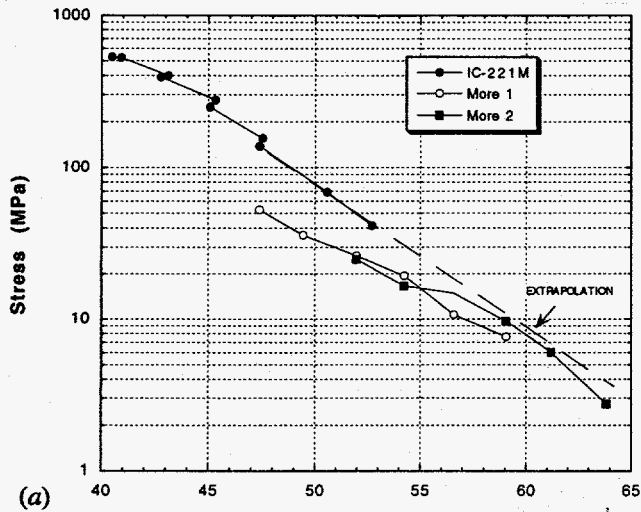


(b)

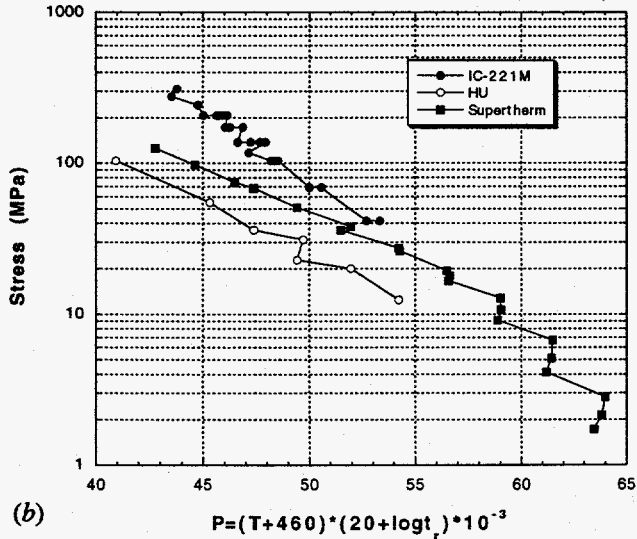


(c)

Figure 7 - Comparison of average tensile properties of wrought Fe<sub>3</sub>Al-based alloys with that of types 422 and 310 stainless steels: (a) yield strength, (b) ultimate tensile strength, and (c) total elongation.



(a)



(b)

Figure 8 - Comparison of creep rupture strength of cast IC-221M with: (a) More 1 and More 2 and (b) HU and SUPERTHERM™.

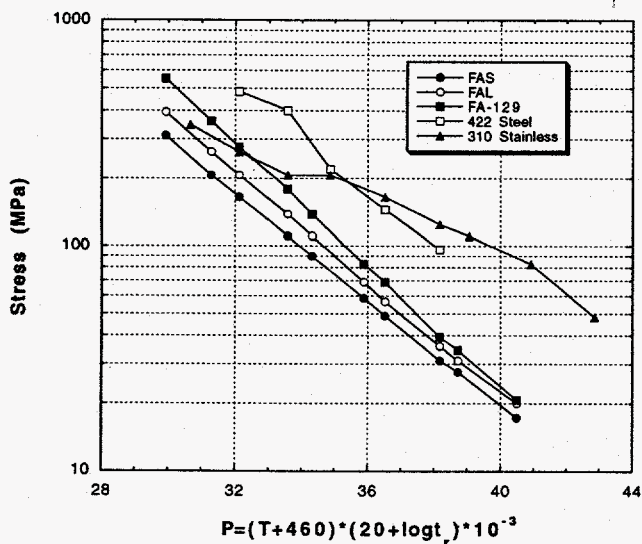
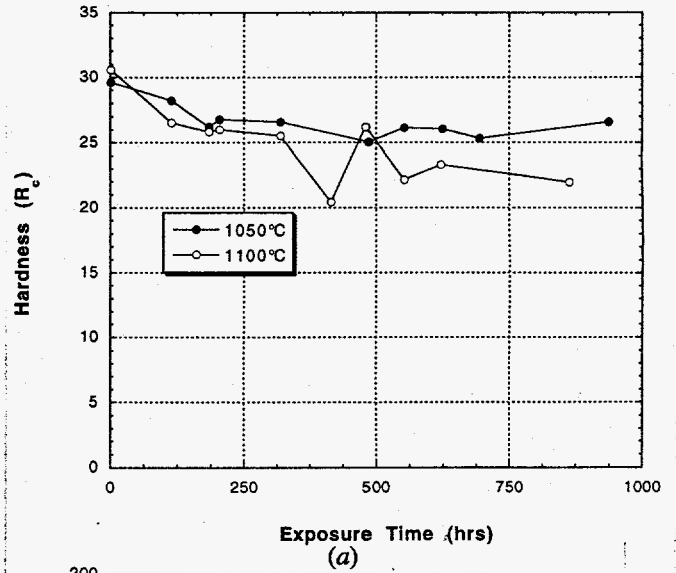
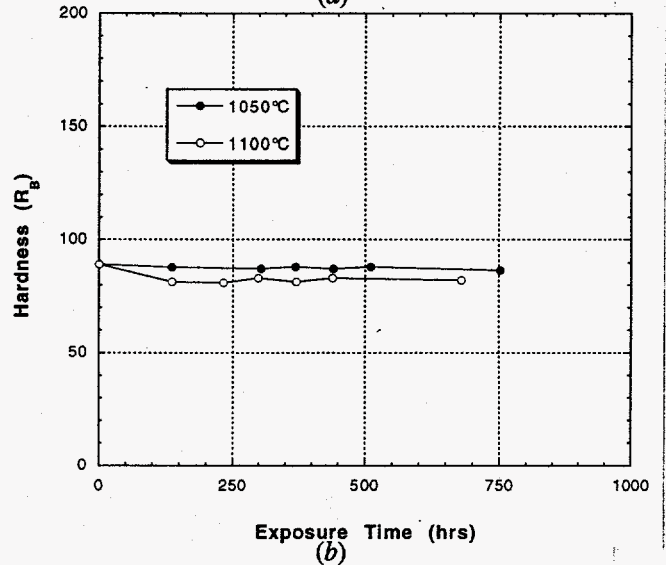


Figure 9 - Comparison of creep rupture properties of wrought Fe<sub>3</sub>Al-based alloys with types 422 and 310 stainless steels.



(a)



(b)

Figure 10 - Effect of thermal aging at 1050 and 1100°C on room-temperature hardness of cast: (a) IC-221M and (b) HU.

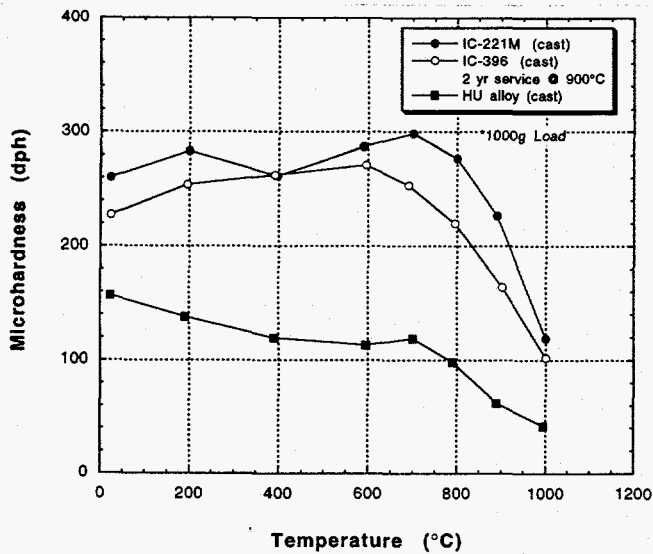


Figure 11 - Effect of two years of thermal exposure at approximately 900°C on hot hardness of cast nickel-aluminide alloy IC-396M (very similar to IC-221M).

carburization, and chlorine environment (see Figures 12-14, respectively), as opposed to commercially available alloys. More details regarding the corrosion properties of Ni<sub>3</sub>Al- and Fe<sub>3</sub>Al-based alloys are available in other papers (29).

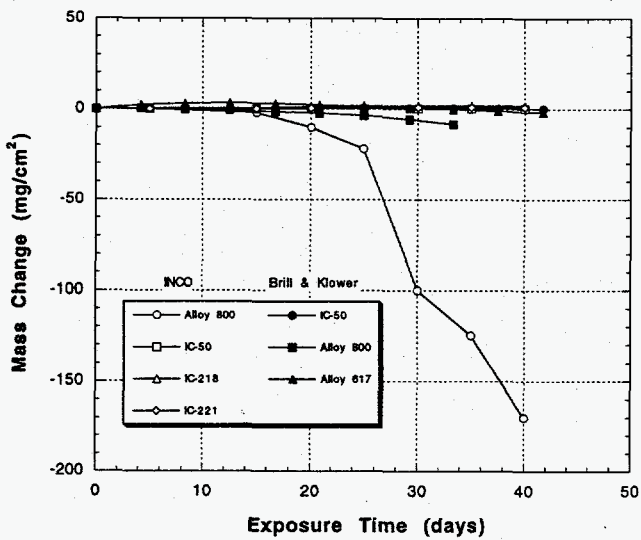


Figure 12 - Oxidation in air - 5% H<sub>2</sub>O vapor at 1100°C of nickel-aluminide and competitive alloys. Data are from INCO Alloys and Brill & Klower.

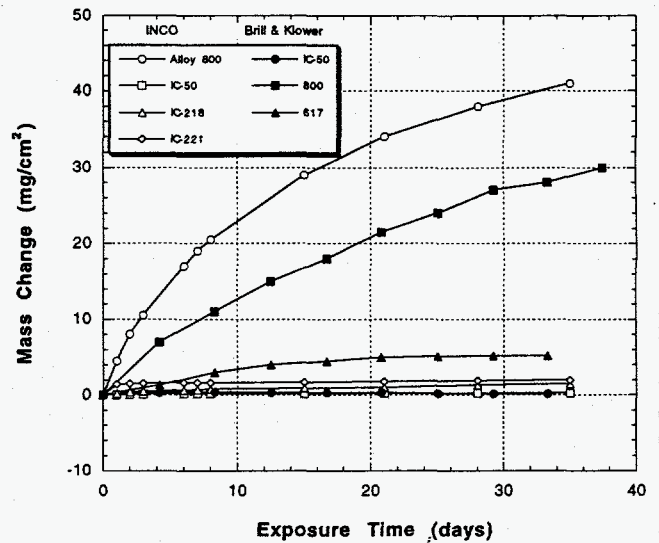


Figure 13 - Carburization in H<sub>2</sub>-1% CH<sub>4</sub> at 1000°C of nickel-aluminide and competitive alloys. Data are from INCO Alloys and Brill & Klower.

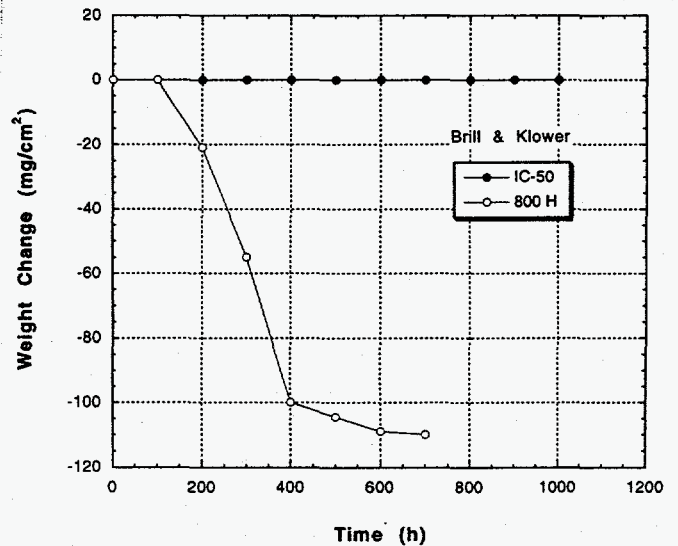


Figure 14 - Corrosion response in Cl<sub>2</sub>/O<sub>2</sub>/Ar at 950°C for nickel-aluminide alloy IC-50 and 800H. Data is from Brill & Klower.

### Applications and Industrial Experience

Both mechanical and corrosion properties have been used to identify the applications for Ni<sub>3</sub>Al- and Fe<sub>3</sub>Al-based alloys. The applications that have been commercialized are briefly described below.

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**Ni<sub>3</sub>Al-Based Alloys.** Because of the high-temperature strength and corrosion properties of the Ni<sub>3</sub>Al-based alloys, furnace fixtures (30) and forging dies have been identified as the two primary application areas.

**Furnace Fixtures.** The furnace fixture applications of Ni<sub>3</sub>Al-based alloy IC-221M include trays and fixtures for carburizing furnaces. In these applications, IC-221M replaces HU alloy and HT type of steels. Trays of IC-221M have operated successfully under production conditions in a batch carburizing furnace for over one and one-half years at Delphi Saginaw (Saginaw, Michigan). Two tray-fixture assemblies have been operating in pusher carburizing furnaces at Delphi Saginaw since January 1996. The IC-221M trays have also been operating successfully in carburizing furnaces at The Timken Company (Canton, Ohio) for the last three months and in a batch heat-treating furnace in an air environment for nearly one year at United Defense LP. Several other applications for trays are currently in the planning stage. Details of the operating experience of some of the trays will be presented by the users at this symposium.

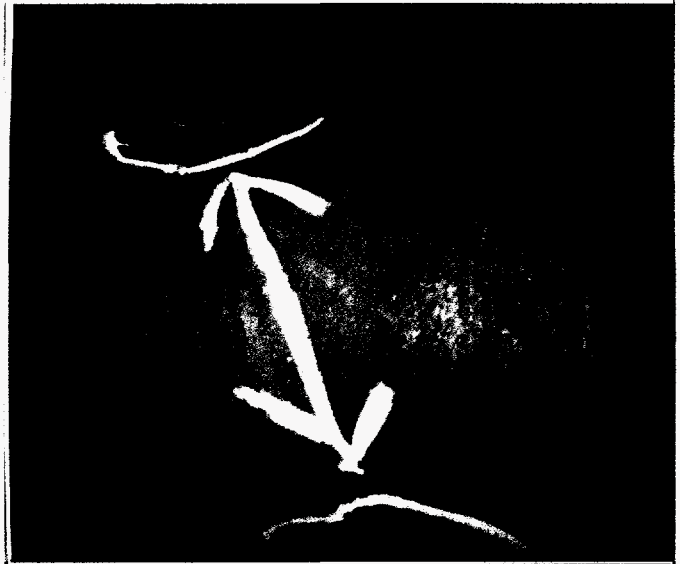
The transfer rolls in a heat-treating furnace in an air environment at Bethlehem Steel Corporation - Burns Harbor Plant (Chesterton, Indiana) are the other application of IC-221M. Two centrifugally cast rolls of IC-221M have operated successfully under production conditions for two years. A photograph comparing the typical damage to a roll made with conventional material versus zero damage to the nickel-aluminide roll is shown in Figure 15. Plans are currently under way to extend the use of 2 rolls to 24 rolls in the furnace at the Burns Harbor Plant by the middle of 1997.

Grate bars, pallet tips, and roll guides are the third furnace fixture application of IC-221M. The grate bars and pallet tips are used for a phosphate ore calcination process. Details of this application will be presented by Orth et al. (31) at this symposium.

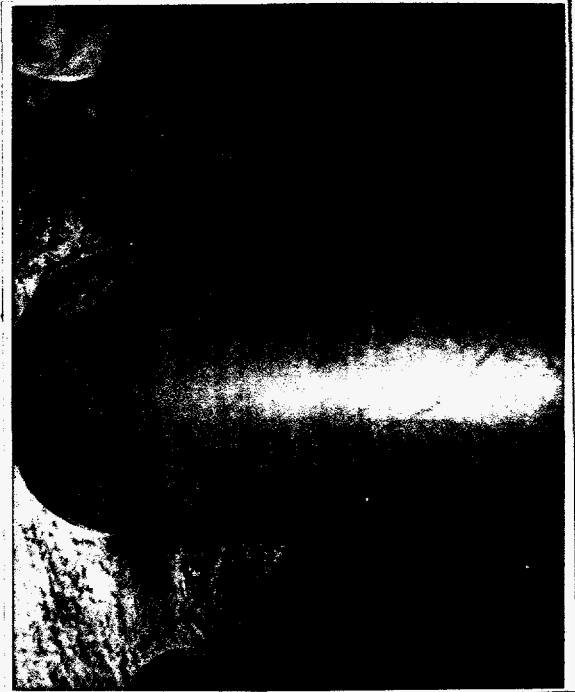
Radiant burner tubes for heating the gas-fired furnaces is an application currently under development. For this application, IC-221M will compete with HU and HK alloy tubes and offer advantages of superior strength, oxidation, and carburization resistance (32).

**Forging Die.** In this application, a sand-cast die block set with part-shape cut using EDM was tested under production conditions at United Defense LP (Anniston, Alabama). The die set was used for hot-forging of 1040 steel bars into a component known as a brake spider. The IC-221M competed with H-13 die blocks, and to date has yielded a factor of eight improvements in life. More details of this application will also be presented by Orth et al. (31).

**Fe<sub>3</sub>Al-Based Alloys.** The low high-temperature strength and limited processability at room temperature have limited the development of broad-based applications of Fe<sub>3</sub>Al-based alloys. The best application of the Fe<sub>3</sub>Al alloy to date is the use in porous gas-metal filters. This application has been developed by PALL Corporation (Cortland, New York), and the details will be presented at this symposium.



(a)



(b)

Figure 15 - Photographs comparing (a) the typical damage to a roll made with conventional material versus (b) zero damage to the nickel-aluminide roll.

The other applications of Fe<sub>3</sub>Al-based alloys are being developed to take advantage of their excellent sulfidation and oxidation resistance. When strength is an issue, the Fe<sub>3</sub>Al-based alloys are being considered as weld overlays or as co-extruded tubes. Efforts are also under way to strengthen the Fe<sub>3</sub>Al-based alloys through oxide dispersion strengthening (ODS). The high-strength ODS alloys based on Fe<sub>3</sub>Al will become available for testing and application development during 1997.

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## Technology Transfer

The ORNL-developed Ni<sub>3</sub>Al-based alloys were the first alloys to go commercial in a big way. The success to the commercialization has resulted from several factors including: (1) development of the data base; (2) prototype testing under production environment; (3) development of the Exo-Melt™ process of air-melting testing equipment commonly available in foundries; (4) choosing a licensee who has the production capabilities, technical expertise, the desire, and need to expand business; (5) ORNL's ability to train the licensee with melting process and overall background of nickel aluminate including welding, which will be discussed in detail at this symposium by Santella (33); (6) ORNL's working closely with the potential users and producers; (7) ORNL's continuing effort to develop the key information that becomes necessary for developing new applications; and (8) ORNL's commitment to assist in microstructural analysis of the components removed from service.

Although only limited applications have been identified for Fe<sub>3</sub>Al-based alloys, the technology transfer path being followed is the same as that described for the Ni<sub>3</sub>Al-based alloys.

### Commercialization Status

One hundred thousand pounds of Ni<sub>3</sub>Al-based alloy IC-221M has been melted, cast, and put into service including the prototype production tests in September 1996. The IC-221M production is projected to go to 200,000 lb during 1997 and to 500,000 lb during 1998. This success in commercialization will act as a spring board for future successes in commercialization of other intermetallics. In addition to the increasing amount of nickel aluminides being used, the nickel aluminides are not strange anymore to a broad range of industries, including steel, aluminum, glass, forest products, metal casting, chemical, metal working, and petrochemical.

### Summary and Conclusions

A comprehensive effort in the development and evaluation under production conditions of Ni<sub>3</sub>Al-based alloys have resulted in a big success in their commercialization. Similar success is expected in the commercialization of Fe<sub>3</sub>Al-based alloys within the next three years. The commercialization success of the Ni<sub>3</sub>Al-based alloy has resulted from the combination of the following factors:

1. Development of data base — This includes physical, mechanical, and corrosion properties.
2. Prototype testing under production conditions — This has been extremely successful for testing of trays in the carburizing and oxidizing furnaces, transfer rolls in a production furnace, and forging dies under production conditions.
3. Development of an economical and safe melting process known as Exo-Melt™ — This development

has allowed the foundries to use conventional equipment in melting nickel-aluminide alloys.

4. Selection of an efficient and productive licensee — United Defense LP, the major licensee for the production of nickel aluminide, has the equipment, expertise, need to expand their business, and the ability to test the prototype components under production conditions. A similar combination did not occur with any of our previous licensees.
5. Technology Transfer effort — The successful transfer of the technology through data, visits, and training of personnel on the production floor has played a very important role in the success of the commercialization effort.
6. Bringing the users and producers together — In this aspect, ORNL not only brought the potential users and producers together, but also enhanced the chances of success by providing data and analysis as needed.
7. Continuing development — ORNL's commitment to further develop the areas needing attention has played an important role in the past and will continue to play a role in future successes. One example of additional development work is higher chemistry range and details of welding procedure for fabricating large components. Another example is analyzing the microstructure of components removed from service and using the results for subsequent application development.

### Acknowledgments

The author thanks C. R. Howell for data plotting; S. C. Deevi from Philip Morris U.S.A. and J. D. Yought for assistance with the Exo-Melt™ process; M. L. Santella for welding process development; Sandusky International for casting and fabrication of roll bodies; Alloy Engineering & Casting Company for casting initial trial trays and assemblies; Delphi Saginaw for testing trays and assemblies in production furnaces; Bethlehem Steel Corporation - Burns Harbor Plant for testing transfer rolls; United Defense LP for producing, marketing, and testing forging dies and trays under production conditions; and FMC Corporation personnel for having the wisdom and desire to put nickel aluminide into trials for calculation applications. The author also thanks C. G. McKamey and C. A. Blue for reviewing the paper, and M. L. Atchley for preparing the manuscript. Note the success of this program would not have been possible without the continuing support from P. S. Sklad and P. Angelini, program managers for the Advanced Industrial Materials Program, and R. R. Judkins, program manager for the Fossil Energy Program.

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