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The Effect of Yttrium Additions on the Tensile Properties and Hardness of an
Iron-Nickel-Chromium Alloy

by

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M.S. Thesis submitted to Iowa State University

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The effect of yttrium additions on the tensile properties
and hardness of an iron-nickel-chromium alloy

by

Mohammed Zafar Shah-Khan

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I. INTRODUCTION

As the world's consumption and cost of oil and natural gas increase every year, nations having nuclear power capability are trying to develop a technology that produces an essentially unlimited amount of economical energy. This technology has been identified as Liquid-Metal Fast-Breeder Reactor (LMFBR) technology. Accordingly, a decision was made by the Energy Research and Development Administration (ERDA), now the Department of Energy (DOE), to commercialize this technology by 1986 (1).

A number of technological areas are being investigated in order to gain a better understanding of the LMFBR technology and to contribute to the data base for the 1986 decision on its commercialization. These areas include: components, physics, chemistry, plant safety, plant experience, fuel recycling, and fuels and materials. In order to make necessary progress in the above mentioned areas, some of the major materials problems must be successfully resolved.

Cladding material for fast reactors must be strong and ductile at the same time, and it must also be compatible with the fuel and coolant environments. Most stainless steels and nickel-based alloys that have been considered are more or less seriously embrittled by irradiation due to the formation of helium gas. At high temperatures, the helium atoms coalesce to form bubbles, which partly concentrate in the grain boundaries. Under creep conditions, most steels rupture at the grain boundaries and therefore, it is clear that the helium bubbles will seriously reduce ductility under these circumstances.

Cladding and duct are the major structural members within the fuel assembly. Typical cladding alloys for various reactor types are zirconium, magnesium, aluminum or stainless steel alloys. The cladding protects the fuel elements from attack by the coolants, contains radioactive fission products, and imparts structural integrity. For LMFBR's the duct is hexagonal or square in shape and provides the means of support and flow control for the fuel pin bundle. The approximate dimensions of fuel pin cladding are 2.5 m long, 0.03-0.04 cm thick and 0.5-0.6 cm in diameter, while the duct is of similar length, 11.5 cm across flats, and 0.30-0.35 cm thick (1).

The current cladding/duct materials for LMFBR's, 20 percent cold-worked 316 stainless steel (CW 316SS), exhibits in-reactor deformation due to swelling and creep, and it is expected to have insufficient creep rupture resistance at the high anticipated operating temperature of about 700°C (1). Also, the expected behavior of CW 316SS has resulted in the use of thicker cladding and duct walls to add strength, and the allowance of large gaps between adjacent fuel assemblies in order to accommodate the expected dimensional changes. Therefore, the possibility of obtaining a peak burnup of over 80 Mwd/kg, with an equilibrium core breeding ratio of about 1.20 (associated with a doubling time of 30 to 40 years) is not present with the current reference fuel assembly design (similar to the Clinch River Breeder Reactor) (1). On the other hand, the generally accepted LMFBR fuel system doubling time and burnup goals are 10 to 15 years and 150 Mwd/kg, respectively (1). Table 1 shows a comparison between the expected

Table 1. Current status and development goals for LMFBR clad/duct materials (from Ref. 1)

Performance Parameter	Reference 316 Current Status	Advanced Alloy Development Goals
Swelling, $\Delta V/V$	18 pct at $2.5 \times 10^{23} \text{ n/cm}^2$	5 pct at goal fluence
In-reactor creep rate, 0.45 kg/mm ² at 650°C	$< 6.2 \times 10^{-7} \text{ h}^{-1}$	$< 2 \times 10^{-7} \text{ h}^{-1}$
Rupture stress, 20,000 h at 650°C	36 kg/mm ²	90 kg/mm ²

performance characteristics of the current cladding/duct material, 20 pct CW 316SS, and the performance goals that were established for the Alloy Development Program. When their performance capabilities are compared, it becomes clear that improved clad/duct materials must be developed in order to achieve these goals.

Recognizing the above demanding conditions, the DOE selected 16 candidate alloys for the Alloy Development Program. Eight of these candidate cladding and duct alloys are for commercial LMFBR applications. Table 2 shows the list of candidate commercial alloys and their candidate developmental alloy counterparts having similar compositions. An Fe-Ni-Cr alloy, designated here as AL1, having the base composition Fe-26% Ni-8% Cr¹ was chosen for this study. It is found that swelling can be decreased in a number of commercially available alloys by increasing the nickel content (2). The explanation for this striking effect of changing the nickel content is not clear and continues to be the subject of much investigation. Additional work (3) revealed that the swelling is further reduced as chromium content is decreased. Also, low chromium content (8%) makes possible large substitutional additions while avoiding phase instability. The high nickel content (26%) and balanced chromium content (8%) should impart excellent resistance to oxidation and carburization at high temperatures. Selected minor element additions such as silicon and

¹Unless otherwise indicated, alloy compositions are given in weight percent.

Table 2. Candidate advanced alloys (from Ref. 1)

Alloy Class	Candidate Commercial Alloy	Candidate Developmental Alloy
Ferritic	HT-9	D57
Solid solution	(316)	D9
Austenitic	310	D11
	330	
Intermediate nickel	A-286	D21
Precipitation Strengthened	M-813	D25
High nickel	Nimonic PE16	D66
Precipitation	Inconel 706	D68
Strengthened	Inconel 716	-
	-	D42

titanium (3-5) can also reduce swelling in a normally high swelling alloy. By maintaining low carbon content (0.035%), complicating carbide effects can be reduced or eliminated. To this base composition a single addition of 0.1% yttrium (rare-earth metal, REM) was made in the present investigation.

Rare-earth metals (REM's) have found wide application in engineering metallurgy (6,7) as a means of reducing the content and neutralizing the detrimental effects of sulfur, oxygen and other harmful impurities present in steels. This beneficial effect of REM's is based on their strong chemical activity, which is indicated by large thermal effects observed when REM's react with sulfur and oxygen to form very stable compounds, such as REM oxides, oxysulfides, and sulfides (8,9). The strong influence of REM's on sulfur in steel, particularly on the removal and spheroidization of sulfur is substantial, as seen in Figure 1 which shows that the free energy of formation of YS is much more negative than FeS. According to a previous study (10), nickel content can be reduced by about 1% in high alloy steels (e.g., 18% Cr - 10% Ni steel) with the addition of REM's without affecting some of the mechanical properties. Gryaznov and Paisov (11) found that the non-metallic inclusions were spheroidal in ingots of 25% Ni-16% Cr steel with REM additions. The size of the inclusions was described as point size inclusions and single large globules. It was experimentally shown (12) that REM's are not carbide formers in iron alloys. They lower the solubility of carbon in the iron and cause

Figure 1. Free energies of formation for iron, yttrium and cerium carbides, nitrides and sulfides as a function of temperature (Refs. 8 and 13)

graphite formation in the iron alloys. Also, it was shown that REM's narrow the gamma-iron region.

In the previous studies, investigators have expressed divergent views concerning the behavior of REM's in steels of widely varying compositions. Most of the information stems from the east European countries, which may be due to the existence of rich sources of raw materials suitable for the production of the REM's. One also finds contradictory data, in different publications, regarding the influence of REM's on the mechanical properties of different steels. All investigators agree, however, that the strength characteristics of steel are not affected by the REM additions (14-23). On the other hand, a number of beneficial effects on the mechanical properties of various steels caused by the addition of REM's are well-documented (21-27). Therefore, the aim of the present study was to investigate the mechanical properties of the developmental alloy AL1 and the effect on these properties of 0.1 percent yttrium (REM) addition.

Tensile and hardness tests are widely employed as means of obtaining some measure of the strength and ductility of a material. The present experimental plan included tensile tests and microhardness tests as a function of temperature from room temperature to 800°C on undoped and yttrium-doped AL1 alloys. The tensile tests and microhardness tests as a function of temperature were performed on 30-mil- and 60-mil-thick sheet specimens, respectively. The tensile tests were conducted in an argon atmosphere and the microhardness tests were performed in vacuum. Room temperature microhardness tests were

performed on shoulder sections of some of the tensile-tested specimens. Annealing studies were carried out on the base material by preparing 30-mil thick sheet specimens, annealing at 100, 200, ..., 700 and 800°C for one hour followed by water quench and tensile testing at room temperature. The shoulder sections of the tensile-tested specimens were later used for room temperature microhardness tests. Chemical analyses and optical metallography were performed at various stages during the investigation. Valuable experience was gained by handling and testing small sheet specimens.

II. EXPERIMENTAL PROCEDURE

A. Sample Preparation

The base material tested in this investigation was AL1 alloy (Fe-26% Ni-8% Cr) from a special heat of material provided by the Westinghouse Hanford Company. The material was received in the form of bar, 1 5/16 inches in diameter. The prior heat treatment was 980°C (1800°F) for 1/2 hr followed by a water quench. The ASTM grain size was 5-6 (0.062 mm-0.044 mm corresponding average grain diameters). The supplier's chemical analysis for chromium and nickel of this bar stock is given in Table 3. Optical metallography was performed on the as-received material (Figure 2) and the concentrations of Fe, Ni and Cr in the as-received material were determined by wet chemistry (Table 4).

Three approximately 80 gm slices of the as-received AL1 alloy were taken from the 1 5/16-inch diameter bar. Two of the slices were arc melted and cast in an argon atmosphere without any chemical additions. The third one was doped with 0.1 weight percent yttrium, but otherwise treated in the same way as the undoped material. The lengths of the cast ingots (fingers) were about 4-5 inches. Hereafter, the finger with the yttrium addition will be called "doped" and the fingers without yttrium addition "undoped".

Table 3. Chemical analysis of as-received
AL1 alloy in weight percent

Heat No. K-192	
Element	Wt. Percent
Chromium	8.65
Nickel	25.71

Figure 2. Optical micrograph of as-received Fe-26% Ni-8% Cr AL1 alloy
(a) transverse cross section, electrolytically polished and etched with glyceresia, at 100X, ASTM grain size #6; (b), (c) and (d) longitudinal sections, mechanically polished and etched with oxalic acid, at 100X, 250X and 1000X, respectively

(a)

Figure 2. Continued

Table 4. Chemical analyses of the as-received AL1 alloy

Element	Chemical Analyses		
	Analytical Results		Averages
Fe (portion 6)	59.23%	59.27%	59.25%
Fe (portion 7)	58.78%	58.80%	58.79%
Ni	25.43%	25.32%	25.375%
Cr	8.32%	8.31%	8.315%

Optical metallography was performed on cuts taken from the doped and undoped fingers in the as-cast condition and following a homogenizing anneal, as shown in Figure 3. The homogenizing anneal was carried out at 1150°C in vacuum at 10^{-6} torr for 16-18 hrs. Amounts of Fe, Ni, Cr and Y were determined by wet chemistry on fingers in as-cast and as-homogenized conditions (Tables 5 and 6). Lengths suitable for rolling were cut from these fingers. A low-speed cutting wheel was used for this purpose. These samples were about 1.0 to 1.5 inches long and had a elliptical cross section. These were first pressed to get flat surfaces for rolling and then rolled to 60-mil thick sheets for microhardness tests and to 30-mil thick sheets for tensile tests. All the above reductions involved cold rolling and intermediate anneals at 1050°C in argon for 30 mins. For intermediate anneals, the samples were sealed in quartz with argon inside at 1/4 atmosphere.

B. Tensile Specimens

Tensile test specimens from the 30-mil sheets were stamped out parallel to the rolling direction. Prior to stamping, the sheet samples were annealed at 1050°C in argon for 30 mins. The stamping was carried out by making use of a special punch and die set (Figure 4). After stamping, the test specimen had a gage section 0.03 inches thick

Figure 3. Optical micrograph of AL1 alloy electrolytically polished and etched with glyceric acid at 100X (a) as-cast, undoped (b) as-cast, doped (c) as-homogenize annealed, undoped (d) as-homogenize annealed, doped

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(c)

(d)

Figure 3. Continued

Table 5. Chemical analyses on as-cast and as-homogenize annealed undoped AL1 alloy

Chemical Analyses (as-cast)				
Element		Analytical Results		Averages
AL1	Fe	59.49%	59.26%	59.375%
	Ni	25.43%	25.26%	25.345%
	Cr	8.28%	8.28%	8.28%
Chemical Analyses (as homogenize annealed)				
Element		Analytical Results		Averages
AL1	Fe	59.70%	59.81%	59.755%
	Ni	25.56%	25.57%	25.565%
	Cr	7.84%	7.94%	7.89%

Table 6. Chemical analyses of as-cast and as-homogenize annealed doped AL1 alloy

Chemical Analyses (as-cast)				
Element		Analytical Results		Averages
AL1Y	Fe	59.18%	59.11%	59.145%
	Ni	25.39%	25.46%	25.425%
	Cr	8.26%	8.19%	8.225%
	Y	0.13%	-	0.13%
Chemical Analyses (as homogenize annealed)				
Element		Analytical Results		Averages
AL1Y	Fe	59.27%	59.33%	59.30%
	Ni	25.49%	25.48%	25.485%
	Cr	8.33%	8.29%	8.31%
	Y	0.10%	-	0.10%

Figure 4. Punch and die set

by 0.06 inches wide by 0.75 inches long. The total length of the tensile specimen was 1.6 inches and the width at the shoulder region was 0.195 inch. The final dimensions of the test specimen were as shown in Figure 5. Holes, 63 mils in diameter, were drilled in the shoulder sections of the test specimen to accommodate 62.5-mil-diameter pins in the grips. Polishing was performed on a 600 grit special silicon carbide paper to get rid of any sharp edges present after the stamping and drilling processes. Then a final anneal at 1050°C in argon for 30 mins followed by air cooling was given to these test specimens, thus making them ready for tensile tests.

For annealing studies on undoped material, the test specimens were prepared in the same way as before but one hour anneals at 100, 200, ..., 700, and 800°C were given to these specimens (two at each temperature) followed by water quenching prior to the room temperature tests. These anneals and quenchings were performed with specimens sealed in quartz containing argon.

C. Tensile Test Apparatus

All of the tensile tests were performed on a screw-driven Instron Model TT-C-L testing machine. This instrument included a load frame and control consoles for specimen loading and furnace temperature control as separate assemblies.

The tensile capsule unit consisted of a container and a pull-rod assembly. The container was constructed of a corrosion-resistant alloy to withstand oxidation and prevent collapse while being evacuated at

Figure 5. Dimensional view of the 30 mil thick sheet tensile specimen

the high temperatures. It had vacuum fittings and a vacuum shut-off valve, line for air cooling, and vacuum seals for the specimen thermocouple. The container also held the bottom static O-ring and a polished bushing at the top, against which the O-ring attached to the upper pull-rod slides.

The pull-rod assembly was made of high-temperature stainless steel. The strength of the assembly exceeded 1500 pounds tensile force at a temperature of 1200°C (2200°F). Pin-type grips were used to grip the test specimen.

Specimen heating was accomplished by an electrically heated furnace. The furnace had three zones, top, center, and bottom. The windings, on a refractory tube, were Kanthal. Each zone had a separate variac control. Furnace temperature and specimen temperature were regulated by a temperature controller through two thermocouples. One thermocouple (Pt-Pt 10% Rh), which was positioned inside the furnace, controlled the furnace temperature and the other (chromel-alumel), which was positioned near the test specimen, monitored the specimen temperature. These two thermocouples were positioned in this way in the system so as to give minimum thermal relaxation time constant.

D. Tensile Test Experiments

Tensile tests were performed from room temperature to 800°C. The crosshead speed was set at 0.1 in./min, which corresponded to a nominal strain rate of 0.22×10^{-2} per sec. The chart speed was set at

5 in./min. Two or three tests were performed at each temperature. The load cell was calibrated before and for each test.

A special jig, Figure 6, was fabricated to hold the grip while the test specimen was being inserted into it. The specimen was inside the grips for a length of about 1/4 inch at each end, and fastened inside the grips by means of four screws at each end. This arrangement prevented slipping in the grips and resulted in all deformation produced within the 0.75-inch gage length.

For room temperature tests, the specimen with the grips was fixed to the upper pull-rod by means of a screw passing through the jaws and the center of the upper grip. The crosshead was moved up with the lower pull-rod pinned to it. Then the lower grip was screwed into the jaws of the lower pull-rod. All the room-temperature tests were carried out in air.

For high temperature tests, the specimen with grips was screwed in both the upper and lower pull-rods by making use of a separate hanger. Then the whole load train was inserted into the vacuum jacket. The vacuum jacket and the load train were as shown in Figures 7 and 8. The vacuum jacket with the load train fastened in it was passed through the core of the furnace and the whole system was raised and installed on the crosshead. A vacuum was pulled in the vacuum jacket and then argon was back-filled into the vacuum jacket. Thus, the test specimen was allowed to soak in argon during the heat-up and the actual test. The system was heated up and when the test temperature reached a steady state the crosshead was moved up and the upper pull-rod was

Figure 6. Fixture to hold the grips and specimen

Figure 7. Load frame and vacuum jacket with specimen pull-rod assembly inside

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Figure 8. Load frame and the specimen pull-rod assembly

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pinned into the load-cell coupling. This procedure allowed the calibration of the machine to be undisturbed. The whole tensile test set-up was as shown in Figure 9. The furnace was so adjusted along the length of the vacuum jacket that when the crosshead was not moving and the system was ready for test, the specimen position was half-way along the furnace length. This set-up gave a dependable temperature reading from the independent thermocouple similar to the temperature recorded on the controller. In this way, the tensile tests were carried out by the downward movement of the crosshead when the specimen was kept in an argon atmosphere. After each test, argon flow was stopped and the specimen was allowed to cool down inside the jacket in vacuum. The times to reach each test temperature were recorded and were as shown in Tables 9 and 10.

E. Hardness Specimens

Specimens 1.8 cm long by 0.4 cm wide were cut out parallel to the rolling direction from the 60-mil-thick sheets on a low-speed cutting wheel. The specimens were then given a final anneal at 1050°C in argon for 30 mins followed by air cooling. The indentation surfaces were prepared by grinding and polishing according to the ASTM standards for diamond pyramid hardness testing (28).

F. Hardness Test Apparatus

Microhardness tests as a function of temperature on 60-mil-thick sheet specimens were performed on a hardness tester constructed based mainly on the design supplied by Oak Ridge National Laboratory (29).

Figure 9. Load frame and the complete high temperature test assembly

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Table 7. All undoped tensile and hardness data

Tensile Test (°C) Temperature	Time to Reach Test Temp. (mins.)	Hardness ^a (DPH)	Ultimate Tensile Stress (kg/mm ²)	Yield Stress (kg/mm ²)	Uniform Strain (%)	Fracture Strain (%)
24 (R.T.) ^b	-	171.0 ± 6.2	63.4 ± 0.4	24.6 ± 0.3	45.6 ± 0.2	47.5 ± 0.6
100	38	171.6 ± 4.8	57.6 ± 0.4	21.2 ± 0.8	43.7 ± 0.8	45.1 ± 1.0
200	58	167.8 ± 6.4	54.0 ± 0.4	20.1 ± 1.3	41.6 ± 1.0	42.6 ± 1.0
300	70	173.7 ± 4.5	53.6 ± 1.1	19.5 ± 1.5	45.2 ± 2.7	46.7 ± 2.9
400	76	176.2 ± 1.8	51.9 ± 1.9	17.9 ± 0.4	52.0 ± 0.6	53.2 ± 0.4
500	93	168.3 ± 5.9	50.6 ± 0.1	18.7 ± 1.3	49.6 ± 1.6	50.6 ± 1.9
600	108	208.2 ± 1.8	49.1 ± 0.9	21.1 ± 0.5	48.9 ± 0.6	50.2 ± 0.5
700	188	298.3 ± 6.2	52.0 ± 1.0	35.8 ± 2.5	25.4 ± 0.8	36.2 ± 6.5
800	200	350.3 ± 6.9	35.3 ± 0.7	31.7 ± 1.8	2.4 ± 0.1	56.8 ± 5.1

^aThese are the room temperature hardness values. Hardness tests were performed on the shoulder sections of the tensile tested samples.

^bTest performed in air.

Table 8.. AL1 doped with 0.1% Y tensile and hardness data

Tensile Test (°C) Temperature	Time to Reach Test Temp. (mins.)	Hardness ^a (DPH)	Ultimate Tensile Stress (kg/mm ²)	Yield Stress (kg/mm ²)	Uniform Strain (%)	Fracture Strain (%)
24 (R.T.) ^b	-	179.5 ± 5.7	61.3 ± 1.8	22.8 ± 0.5	44.1 ± 0.4	45.1 ± 0.1
100	42	169.1 ± 1.6	57.1 ± 0.2	20.2 ± 0.6	41.4 ± 0.7	42.7 ± 0.9
238	65	180.7 ± 7.9	54.0 ± 0.4	18.1 ± 0.2	43.1 ± 1.3	44.2 ± 1.4
300	68	178.8 ± 1.1 ^c	53.8 ± 0.2	17.1 ± 0.1	47.8 ± 0.4	49.3 ± 0.6
410	70	172.4 ± 3.8	50.8 ± 0.3	16.8 ± 0.2	46.8 ± 0.3	47.8 ± 0.3
498	95	176.7 ± 2.5	50.1 ± 0.6	16.9 ± 0.3	48.6 ± 0.5	49.6 ± 0.6
598	105	201.6 ± 2.6	48.3 ± 0.5	19.1 ± 0.4	47.2 ± 0.5	48.2 ± 0.4
698	196	274.5 ± 6.2	52.1 ± 0.6	35.1 ± 1.0	22.0 ± 0.8	24.9 ± 0.3
798	194	363.8 ± 3.6	35.9 ± 0.2	35.0 ± 0.1	2.8 ± 0.2	48.5 ± 2.9

^aThese are the room temperature hardness values. Hardness tests were performed on the shoulder sections of the tensile tested samples.

^bTest performed in air.

^cValue from edge surface of the shoulder section.

Figure 10 shows the schematic diagram of the elevated temperature microhardness tester. Tests on this hardness tester can be conducted under vacuum or inert atmosphere and up to 850°C. The hardness tester comprises two main mechanisms: the translational motion mechanism through which the indenter can be moved in x and y directions, and the push rod drive mechanism which supports the specimen and brings it in contact gradually and smoothly under the predetermined load of the indenter and added weights. The contact and contact-break points between the specimen and the indenter are found by making use of an electrical contact circuit.

The furnace temperature and the specimen temperature were manually controlled, with $\pm 2^\circ$ of the nominal test temperature, through an external autotransformer and were read off a digital temperature indicator. Figure 10 shows the locations of the furnace and specimen thermocouples.

The room temperature microhardness tests were performed on a Tukon hardness tester. These tests were conducted on the shoulder sections of the 30-mil thick tensile tested specimens. The hardness tester consists of an indenter (a 136° diamond pyramid type) and, to place the specimen, a specially designed mechanical stage. The movement of this stage is controlled in two directions by micrometer screws to locate a test area. The stage fits on the elevating screw which can be raised or lowered by a reversible motor through a chain drive and worm gear.

Figure 10. Front view of the elevated temperature microhardness tester

In operation, the sliding stage and the specimen are gently pushed under the indenter and the green button on the front panel is momentarily depressed. The elevating screw motor starts immediately and thereby moves the elevating screw upwards toward the indenter. The specimen contacts the indenter and lifts the loaded beam away from the insulated contact support; thereby further upward motion is stopped instantaneously. After a timed interval, a lifting fork actuated by an eccentric cam and cable automatically raises the indenter away from the specimen while the elevating screw motor lowers the specimen. The indentation cycle is completed when the elevating screw motor stops.

G. Hardness Test Experiment

Microindentation hardness tests were performed from room temperature to 800°C under a test load of 1.224 kg_f (0.120×10^2 N). All the hardness tests as a function of temperature were conducted under vacuum of about 10^{-4} torr. A highly polished, pointed and square-based pyramidal sapphire indenter having included face angles of 136 degrees was used. This test is known as the diamond pyramid hardness or Vickers hardness test. The hardness of a material is expressed as the diamond pyramid hardness (DPH) number. This number is obtained by dividing the applied load in kilograms-force by the surface area of the indentation in square millimeters obtained from the mean of the measured diagonals of the square indentation. The following equation is then used to calculate the hardness:

$$\text{DPH} = P/A = \frac{2P}{d^2} \sin \frac{\theta}{2}$$

where

P = load in kilograms-force

A = surface area of indentation in mm^2

d = mean diagonal length of indentation in mm

and

θ = the included face angle of the indenter = 136 degrees

The procedure for making elevated temperature hardness tests was as follows. With the test specimen under vacuum inside the bell jar and about 1/8 inch from the indenter tip, the furnace was turned on. This initial placement of the specimen from the indenter tip was done using the coarse control in the push-rod drive mechanism. After the temperature reached a steady-state, the fine control in the push-rod drive mechanism was used to raise the specimen against the indenter plus added weights (1.224 kg_f). As mentioned earlier, the contact and contact-break points were found using an electrical contact circuit which switches off and on respectively. The time of application of the full test load was approximately 15 seconds as measured by a stopwatch. The time was measured from the moment the specimen supports the full load to the time the load is released. Subsequent indentations at different test temperatures were made by translating the indenter on a pre-determined course along the specimen using the x and y translational motion mechanism. One test specimen was used for three test temperature hardness measurements. Great care was taken to keep the whole system free of vibrations and shocks. The diagonal lengths of the indentations were measured using a filar eyepiece with a precision better than

0.5 microns.¹ The filar eyepiece was earlier calibrated to a standardized hardness block obtained from the Wilson Instrument Company. A minimum of three to a maximum of eight indentations were used in calculating the hardness values.

Room temperature microhardness tests on the shoulder sections of the 30-mil-thick tensile tested specimens were performed on a Tukon tester. A minimum of three to a maximum of five indentations were made with the specimen mounted in a bakelite cold. The indenter was of the diamond pyramid type having 136 degree face angles. The test load was 1000 kg_f and time of application of the full test load was approximately 22 seconds. The diagonal lengths of the indentation were measured by viewing the indentation through a microscope, which had a built-in eyepiece and the DPH was calculated by using the previously mentioned equation.

To sum up, two sets of hardness experiments were conducted. One on 60-mil thick specimens as function of temperature and another on the shoulder sections of the 30-mil thick tensile tested specimens at room temperature.

¹All measurements of the indentation diagonal lengths were made at room temperature.

III. RESULTS

Tensile specimens of doped and undoped AL1 alloy (Fe-26%Ni-8%Cr) were tested at temperatures ranging from room temperature to 800°C at a constant strain rate of 0.22×10^{-2} per sec. Figures 11-14 illustrate yield stress (YS), ultimate tensile stress (UTS), uniform strain (ϵ_u), and fracture strain (ϵ_f) versus test temperature (T_t) for doped and undoped alloys. The above results are also listed in Tables 7 and 8. For the undoped alloy, the yield stress at room temperature was found to be 24.6 kg/mm^2 , followed by a gradual drop to 17.9 kg/mm^2 at 400°C, indicating the increasing ease with which the material yields with increasing deformation temperature. In the absence of strengthening effects with increasing temperature due to precipitation of any second phase particles that might impede or act as barriers to the motion of slip dislocations, the material will have only the primary barriers that existed at room temperature. Thus, with increase in deformation temperature these primary barriers become less effective (or weaker) in impeding the motion of slip dislocations, resulting in lower values of yield stress. The yield stress showed a gradual upward trend, starting at 400°C, and above 600°C the yield stress increased sharply to a value of 35.8 kg/mm^2 at 700°C. This strengthening as measured by yield stress, must be associated with interactions between moving dislocations and such barriers as solute atoms, precipitates, and pinned dislocations. The yield stress showed a slight decrease at 800°C for the undoped alloy. The addition of 0.1 wt percent yttrium

Figure 11. 0.2 percent yield stress versus test temperature for undoped and doped AL1 alloy

Figure 12. Ultimate tensile stress versus test temperature for undoped and doped ALI alloy

Figure 13. Uniform strain versus test temperature for undoped and doped ALI alloy

Figure 14. Fracture strain versus test temperature for undoped and doped alloy AL1

resulted in slightly lower yield stresses between room temperature and 700°C but the shape of the curve showed no change from the undoped curve. At 800°C the doped alloy showed slightly higher yield stress compared to the undoped alloy.

The ultimate tensile stress, UTS, (Figure 12) of doped and undoped alloys showed nearly no change over the whole test temperature range. The room temperature UTS for the doped alloy (61.3 kg/mm^2) was slightly lower than for the undoped alloy (63.4 kg/mm^2). The UTS reached gradually to a first minimum at 600°C followed by a slight increase between 600°C and 700°C. Above 700°C the UTS dropped fairly rapidly to about 35 kg/mm^2 at 800°C.

The uniform strain, ϵ_u , (Figure 13) for the undoped alloy was relatively insensitive to temperature up to 600°C above which it dropped rapidly to 2.4% at 800°C. There was found to be no significant change in uniform strain values with the addition of 0.1% yttrium when compared with the uniform strain values obtained for undoped alloy. The room temperature uniform strains for doped and undoped alloys were 44.1% and 45.6%, respectively.

The fracture strain (ϵ_f) values for the undoped alloy were slightly higher at most test temperatures (Figure 14). The shapes of the ϵ_f versus test temperature curves for doped and undoped alloys were similar. The room temperature fracture strains for doped and undoped alloys were 45.1% and 47.5%, respectively. As can be seen in Figure 14, the fracture strain dropped to 36.2% for undoped alloy and to 24.9% for doped alloy at 700°C. Above 700°C, the fracture strain increased to

48.5% and 56.8% for doped and undoped alloys at 800°C, respectively. Figures 15-17 illustrate stress-strain plots obtained for undoped and doped alloys at room temperature, 400°C and 800°C tests.

Room temperature microhardness tests were performed on the shoulder sections of the tensile tested specimens of doped and undoped alloys. Figure 18 illustrates the room temperature hardness for the undoped and doped alloys. The hardness was found to be fairly constant from room temperature to 500°C. Above 500°C there was a rapid increase in hardness, reaching 350.3 DPH at 800°C. The hardness curve for the doped alloy followed the same trend as found for the undoped alloy. The room temperature hardness for the doped and undoped alloys were 179 DPH and 171 DPH, respectively. Tables 7 and 8 list the hardness values for undoped and doped alloys.

Optical metallography was performed on the shoulder section of the elevated temperature tensile tested specimens. Both the edge-on and flat-on surfaces were used for this purpose and Figures 19 and 20 illustrate the optical micrographs obtained from the 100°, 400° and 800°C tested shoulder sections for undoped and doped alloys, respectively. It is evident from the micrographs that there does not exist any change in the distribution of the second phase.

Annealing studies were carried out on undoped alloy to see the effect of time at temperature on the results obtained from previous tensile tests. Tensile tests, on undoped specimens held for one hour at T_A followed by quenching into water, were performed at room temperature in air. The annealing temperatures, T_A , were 100°C, 200°C, ..., 700°C and 800°C. Yield stress, YS, ultimate tensile stress,

Figure 15. Stress-strain curve at room temperature for undoped and doped AL1 alloy

Figure 16. Stress-strain curve at 400°C and 410°C for undoped and doped AL1 alloy, respectively

Figure 17. Stress-strain curve at 800°C and 798°C for undoped and doped AL1 alloy, respectively

Figure 18. Room temperature hardness versus tensile test temperature for tests performed on the shoulder sections of the tensile tested specimens for undoped and doped ALI alloy

Figure 19. Optical micrograph of the shoulder sections of the undoped tensile tested specimens, electrolytically polished and etched with oxalic acid at 500X. (a) and (d) tested at 100°C, flat-on and edge-on view respectively (b) and (e) tested at 400°C, flat-on and edge-on view respectively (c) and (f) tested at 800°C, flat-on and edge-on view respectively

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Figure 19. Continued

Figure 20. Optical micrograph of the shoulder sections of the doped tensile tested specimens, electrolytically polished and etched with oxalic acid at 500X. (a) and (d) tested at 100°C, flat-on and edge-on view respectively (b) and (e) tested at 410°C, flat-on and edge-on view respectively (c) and (f) tested at 798°C, flat-on and edge-on view respectively

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Figure 20. Continued

UTS, uniform strain, ϵ_u , and fracture strain, ϵ_f , were determined as a function of annealing temperatures and plotted as shown in Figures 21-24. Table 9 also lists the values of the above properties. The room temperature yield stress (Figure 21) was fairly constant between the annealing temperatures 100°C and 500°C. With T_A above 500°C, the room temperature yield stress increased rapidly and the 800°C anneal resulted in doubling the room temperatures yield stress. The effect of T_A on room temperature ultimate tensile stress (Figure 22) was significant at T_A greater than 500°C, but not as pronounced as in the case of room temperature yield stress. The increase in room temperature ultimate tensile stress due to the 800°C anneal was about 1 1/2 times that obtained by 100°C anneal.

The effect of annealing temperatures on the uniform strain and fracture strain can be seen from Figures 23 and 24, respectively. Both the uniform strain and fracture strain decrease rapidly with annealing temperatures above 500°C. With T_A lower than 500°C, the uniform strain and fracture strain are fairly constant.

Room temperature microhardness tests were performed on the shoulder sections of tensile specimens used in the annealing studies. Specimens annealed at 100°, 400°, 600° and 800°C were used for this purpose. Figure 25 shows plots of room temperature ultimate tensile stress and room temperature hardness as a function of annealing temperature.

It was shown in previous studies (30) that a general relationship exists between the hardness (H) and the ultimate tensile stress (σ_{UTS}) for a given material. The relation is given as:

Figure 21. 0.2 percent yield stress as a function of annealing temperature for undoped AL1 alloy

Figure 22. Ultimate tensile stress as a function of annealing temperature for undoped ALI alloy

Figure 23. Uniform strain as a function of annealing temperature for undoped AL1 alloy

Figure 24. Fracture strain as a function of annealing temperature for undoped AL1 alloy

Table 9. Undoped AL1 alloy, tensile and hardness results from annealing studies

Annealing Temperature °C	Room Temperature Yield Stress, kg/mm ²	Room Temperature Ultimate Tensile Stress, kg/mm ²	Room Temperature Uniform Strain Percent	Room Temperature Fracture Strain Percent	Room Temperature ^a Hardness, kg/mm ²
100	26.0 ± 1.1	64.1 ± 1.7	46.9 ± 0.0	48.3 ± 0.2	176.6 ± 3.3
200	26.1 ± 2.6	65.6 ± 1.7	47.4 ± 1.5	48.4 ± 1.1	
300	27.2 ± 0.1	66.6 ± 0.3	46.3 ± 0.7	47.2 ± 0.4	
400	23.3 ± 0.2	60.7 ± 1.1	47.2 ± 0.2	48.4 ± 0.4	178.8 ± 7.4
500	25.4 ± 0.2	63.6 ± 0.0	46.1 ± 2.4	47.1 ± 1.3	
600	32.0 ± 1.1	70.1 ± 0.5	45.8 ± 1.0	46.6 ± 1.0	224.2 ± 4.0
700	46.2 ± 0.4	85.4 ± 0.1	36.8 ± 0.3	37.8 ± 0.3	
800	52.0 ± 0.2	96.2 ± 0.3	28.3 ± 1.1	29.4 ± 1.1	319.1 ± 9.24

^aTests performed on the shoulder sections of the tensile tested specimens.

Figure 25. Ultimate tensile stress and hardness as a function of annealing temperature for undoped AL1 alloy

$$H = C \sigma_{UTS}$$

where C is a constant known as the constraint factor for the hardness test. The value of C varies between 2.57 and 3.1, depending upon the behavior of the material to indentation, whether in a rigid-plastic manner or plastic-elastic manner or plastic-rigid manner (31,32). Tabor (33) and Cahoon (34), using different relations, found the value of C to be 2.9 and 3.0 ± 0.1 for steel, respectively. In this study, the values of C were in the range 2.7 to 3.3 which gave an average $C = 3.0$, when the above relation was used. Table 9 lists the hardness values obtained in the above tests. Figure 26 shows the optical micrographs obtained from head sections of specimens annealed at 100, 400, 600 and 800°C followed by water quench.

Effect of temperature on the indentation hardness for undoped and doped AL1 alloy was found by conducting tests on 60-mil thick specimens. Figure 27 illustrates the plots of hardness in DPH as a function of temperature. The tests were performed in vacuum using a 136 degree sapphire pyramid type indenter with a test load of 1.224 kg_f, and the time to reach each test temperature was approximately one hour.

Room temperature hardness values for undoped and doped AL1 alloys were found to be 168.5 DPH and 174.6 DPH, respectively. It is evident from Figure 27, there is no significant change in hardness from room temperature to 600°C for undoped and doped alloys while the hardness generally decreases in the same temperature range. The hardness for undoped and doped alloys decreased to 121.7 DPH and 123.2 DPH, respectively at 600°C. Above 600°C both the alloys showed a

Figure 26. Optical micrograph of the shoulder sections of the undoped tensile tested specimens, electrolytically polished and etched with oxalic acid, at 100X (a) annealed at 100°C (b) annealed at 400°C (c) annealed at 600°C (d) annealed at 800°C, for one hour followed by water quench

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Figure 27. Indentation hardness as a function of temperature for undoped and doped Al1 alloy

strengthening effect with hardness increasing rapidly above 700°C.

The doped alloy appeared to be stronger at higher temperatures with a hardness of 188.5 DPH at 800°C, compared to the hardness of 175.8 DPH obtained for undoped alloy at the same temperature. Table 10 lists the hardness values obtained in the above tests.

Table 10. Hot-hardness results for undoped and doped alloys

Test Temperature, T_t ($^{\circ}\text{C}$)	Hot Hardness, H (DPH)	
	Undoped	Doped
25	168.5 ± 2.0	174.6 ± 1.4
100, 150	164.0 ± 2.6	158.3 ± 1.8
200	144.6 ± 2.5	145.7 ± 1.7
300	136.7 ± 2.1	140.2 ± 1.2
400	144.2 ± 1.0	132.5 ± 1.8
500	139.6 ± 1.0	132.7 ± 2.0
600	121.7 ± 1.1	123.2 ± 2.2
700	125.9 ± 0.5	134.0 ± 1.6
800	175.8 ± 1.6	188.5 ± 1.2

IV. DISCUSSION

The results obtained in the present study concerning the effect of yttrium addition on the mechanical properties of AL1 alloy agree very well with results obtained by previous investigators (35,36) working with yttrium and other rare-earth metals in steels. Gulyaev and Ul'yanin (35) obtained no change in the strength and ductility of an Fe-1% Cr-0.4% Mn steel with addition of 0.05%-0.30% Ce, La, Nd and Pr. Pridantsev, Kondrat'ev and Ivanov (36) working with an austenitic (Fe-18% Cr-12% Mn-5% Ni) steel containing 1% Nb and 0.45-0.52% N found practically no effect on the tensile strength or elongation at temperatures ranging from 20°C to 1100°C with addition of 0.003-0.011% Ce. Although other information exists about the beneficial effects of rare-earth metals in steels, previous results are contradictory concerning the effect of rare earths on the mechanical properties. Gryaznov and Paisov (27) indicate an increase in strength but a decrease in ductility with the addition of 0.2% Ce to Fe-26% Ni-15% Cr steel.

In the present investigation as shown in Figures 11-14 it was found that there exists no significant change in yield stress, ultimate tensile stress, uniform strain, fracture strain and microindentation hardness as a function of temperature with the addition of 0.1 wt % yttrium to the base AL1 alloy. Above 700°C, there was a slight improvement in the yield stress and hardness with yttrium addition, as seen in Figures 11 and 26, respectively.

The strengthening effect noticed in the undoped and doped ALL alloy above 600°C can be explained by any one of a number of mechanisms and this creates a major problem in deciding which mechanisms are important in this experimental situation. Strengthening can be associated with interactions between moving dislocations and such barriers as solute atoms, pinned dislocations and precipitates.

From the start it was known (not definitely) that ALL alloy is a precipitation hardening alloy. Also, the presence of nickel, aluminum and titanium in high amounts might lead to some kind of precipitation at higher temperatures and longer times of exposure. Therefore, with the presence of the above condition in this investigation, the precipitation of γ' phase- $\text{Ni}_3(\text{Ti}, \text{Al})$ is suspected of causing the strength to increase at temperatures above 600°C. In support of the above suspicion, Orr (37) indicates that with carbon content 0.03% or lower, the major portion of the total titanium content is retained in solution when cooled from solution treatment temperatures. This retained titanium and also possibly aluminum may precipitate out as $\text{Ni}_3(\text{Ti}, \text{Al})$ during reheating at 600-700°C, thus causing the strengthening in the high temperature range. This strengthening might be causing the reduction in ductility as found in the annealing studies (Figures 23 and 24).

The effect of temperature on fracture strain (Figure 14) is interesting and worth mentioning. It was found that with increase in time and temperature a ductility maximum was observed, followed by a ductility minimum and a second ductility rise. This type of

observation was also obtained by Broomfield (38) working with a similar LMFBR candidate alloy, Nimonic PE16. The ductility minimum is explained to be due to diffusion strain effects. A possible interpretation of diffusion strain is that grains are elongated by diffusion of atoms, while vacancies form as a counter-current on the grain boundaries aligned parallel to the stress axis and reducing the section under stress. Grain boundary carbides are shown to be taking part in the diffusion strain effects. The recovery of ductility with increasing temperature is explained to be due to the relief of stress on grain boundary carbides.

V. SUMMARY

The following summary can be drawn from this investigation.

1. The addition of 0.1 percent yttrium does not significantly change the mechanical properties of the AL1 alloy with temperature. However, the yttrium-doped samples did show a slight increase in yield stress and hardness for tests above 700°C.
2. The room temperature strength of the undoped AL1 alloy increases upon annealing at temperatures above 600°C. The source of this strengthening may be associated with the precipitation of γ' -Ni₃(Ti,Al) phase.
3. The room temperature uniform and fracture strains of the undoped AL1 alloy decrease upon annealing at temperatures above 600°C.

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