THE STRUCTURE AND PROPERTIES OF A DISPERSION-STRENGTHENED Fe-Cr-Al ALLOY,
INCOLOY ALLOY MA 956E

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ABSTRACT

The structure and properties of a newly developed, dispersion-strengthened,
ferritic Fe-Cr-Al alloy have been examined. Designated INCOLOY alloy
MA 956E, the alloy was prepared by the mechanical alloying technique utilizing
Y_2O_3 as a dispersoid. In the cold-rolled plus annealed condition, the
alloy possesses a coarse-elongated grain structure similar to that observed
in dispersion-strengthened nickel-base alloys. The stress rupture properties
at 2000°F (1093°C) for 0.06 inch thick sheet exceed reported values for TD
nickel. The stress rupture curves also show an exceedingly flat slope,
indicating good long time stability.

Room temperature bend tests indicate the sheet product has excellent
fabricability in that bend angles greater than 150° were obtained (about a
diameter equal to twice the sheet thickness).

Cyclic oxidation, burner-rig and carburization tests all showed only a
small weight gain after long time exposures. Metallographic examination of
the exposed specimens showed no evidence of subsurface attack.

The properties of INCOLOY alloy MA 956E measured in this investigation,
together with the high melting point, low density and low thermal expansion
coefficient that are characteristic of a ferritic composition suggest the
alloy would have good potential for use in a variety of high temperature
sheet applications.

Introduction

One of the areas of high temperature materials research that has received considerable attention, is the utilization of dispersion strengthening to improve the elevated temperature strength of various alloys. This research has been aimed primarily at nickel-base alloys, and has resulted in the development of such dispersion strengthened materials as TD nickel, TDNiCr and IN-853(1).

While a significant effort has been expended on dispersion-strengthened nickel-base alloys, relatively little work has been done on iron-base, ferritic materials for high temperature use. For high temperature applications such as gas turbine components a ferritic composition offers certain distinct advantages. The melting point of iron-chromium alloys, for example, is generally over 2700°F (1482°C), well above that of typical nickel-base alloys. The density of a body-centered-cubic, iron-base alloy is at least 10% lower than a face-centered-cubic alloy providing a significant strength-to-weight advantage. Furthermore, ferritic compositions are characterized by a lower thermal expansion coefficient than nickel-base alloys which should be of benefit to the thermal fatigue life.

Dispersion strengthened ferritic alloys have been examined by Oda and Daikoku(2) who observed an increase in the creep rupture strength of a 13CrMoTi and an AISI 406 steel by adding γAl_2O_3 to the prealloyed powders. Allen and Perkins(3) extensively studied a number of alloys such as Fe-15Cr-6Al-2Y which were dispersion strengthened by internal oxidation of the prealloyed powder. They obtained elevated temperature strengths and oxidation resistance similar to that of TDNiCr.

In the present study, the structure and properties of an Fe-20Cr-4.5Al-0.5Ti-0.5Y₂O₃ alloy, designated INCOLOY* alloy MA 956E, have been investigated. The alloy was prepared by the mechanical alloying technique(4) which allows direct incorporation of the oxide dispersoid into the metal matrix via high energy milling of the starting powders. The data presented here were obtained on commercial-size sheet material.

Experimental Details

Materials

The starting powders used to prepare the alloys consisted of elemental iron (-100 mesh), yttria ($\sim$200Å) and a master alloy powder with the approximate composition of Fe-60Cr-15Al-1.5Ti (-60 mesh).

Processing

The blended starting powders were then processed using the mechanical alloying technique described by Benjamin(4). The chemical analysis of the processed powder is given in Table I.

Table I

Chemical Analyses of MA 956E Alloy

Fe	Cr	Al	Ti	Y₂O₃	O*	N
Bal.	19.1	4.6	0.50	0.50	0.26	0.042

*Total oxygen content including dispersoid.

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Following attrition the powder was sealed in a mild steel can and extruded
to a rectangular bar. After removing the steel can, the alloy was hot
rolled to plate and subsequently cold rolled to 0.06 inch thick sheet
material. The sheet product was annealed at 2425°F (1330°C) and surface
ground prior to testing.

Results and Discussion

Microstructure

The grain structure of dispersion strengthened nickel-base alloys produced
by mechanical alloying is generally characterized by coarse, elongated
grains(4,5). This type of grain structure is developed during a high
temperature annealing treatment by a process resembling secondary recrystal-
lization(5). It has been shown that the presence of the coarse elongated
grains is necessary for the attainment of good high temperature stress
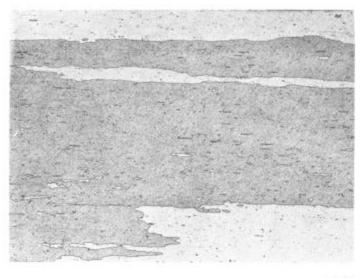rupture properties(5,6).

Microstructural examination of the MA 956E alloy revealed a similar
grain structure to that which occurs in dispersion-strengthened nickel-base
alloys. The sheet material contains large pancake-shaped grains that are
approximately equiaxed in the plane of the sheet with grain diameters
exceeding 5mm. Figure 1 shows a longitudinal section (cut parallel to the
rolling direction) of the annealed sheet material. The thickness of the
grains is in the range of 0.2-0.5mm, thereby providing aspect ratios of
greater than 10 for both longitudinal and transverse directions in the
sheet plane.

The structure shown in Figure 1 is also characterized by a number of
fine elongated grains contained wholly within the larger grains. These
smaller grains may be remnant areas of misorientation that recrystallize
independently, and can not be accommodated by the sweeping boundary that
produces the larger grains. Similar grain structures have been observed in
dispersion-strengthened nickel-base alloys(7).

The alloy was also examined by electron microscopy to determine the
nature of the internal structure. Figure 2 shows a montage of electron
micrographs taken of a typical area in the sheet material. The bulk of the
structure is relatively free of dislocations although local areas of high
dislocation density can be seen within the narrow grain running from upper
left to lower right. This low dislocation density is also typical of
dispersion strengthened nickel-base alloys following the grain coarsening
annealing treatment.

It is also evident that the structure contains a uniform dispersion of
fine particles. Both electron and x-ray diffraction analyses were made on
extracts of the sheet material in order to determine the nature of the
particles. The results showed the major oxide phase present to be
$3Y_2O_3 \cdot 5Al_2O_3$ along with a minor amount of $Y_2O_3 \cdot Al_2O_3$. In addition to the
oxides, the extracts showed a strong diffraction pattern for Ti(C,N). The
x-ray line broadening of the various phases indicates the $3Y_2O_3 \cdot 5Al_2O_3$ and
$Y_2O_3 \cdot Al_2O_3$ phases have particle sizes below 500Å. The particle size of the
Ti(C,N) particles, however, is generally well above 1000Å. It seems likely,
therefore, that the major portion of the fine dispersion of particles in
Figure 2 is a combination of the two yttria aluminates.

The reaction of Y_2O_3 with aluminum and oxygen to form mixed oxides has
also been found to occur in a dispersion strengthened nickel-base alloy by
Benjamin et al(8). Their results indicate that the formation of the mixed
oxides increases the total volume fraction of dispersoid by a factor of
about 1.75 over the initial Y_2O_3 addition. Once formed, however, the mixed
oxides were extremely stable and showed no significant increase in particle

100X

FIGURE 1

LONGITUDINAL SECTION OF MA 956E SHEET SPECIMEN SHOWING
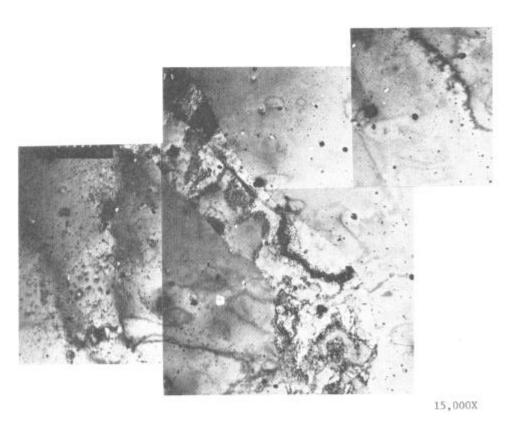ELONGATED GRAIN STRUCTURE

15,000X

FIGURE 2

ELECTRON-MICROGRAPH MONTAGE OF STRUCTURE IN MA 956E

size after exposures of 1000 hours at 1900°F (1038°C), and only a minor
increase after 50 hours at 2400°F (1316°C).

Stress Rupture Properties

The stress rupture properties were determined at 1800°F (982°C) and
2000°F (1093°C) and are shown on a log stress vs. log time plot in Figure
3. At 2000°F (1093°C) the longitudinal life exceeded 1500 hours at 8.5 ksi
(58.6 MPa) and the transverse life was greater than 2000 hours at 8 ksi
(55.2 MPa). The stress rupture properties of MA 956E show a high degree of
isotropy and compare favorably with published data for stress relieved TD
nickel sheet(9), also shown in Figure 3.

The stress rupture curves of dispersion strengthened alloys are generally
characterized by relatively shallow slopes. In the case of MA 956E the
curves are exceedingly shallow (slopes of approximately -0.03) and are
noticeably flatter than that of TD nickel.

Tensile Properties

The ultimate tensile strength was determined at various temperatures in
both the longitudinal and transverse sheet directions. The results given
in Table II also show a high degree of isotropy in strength over the temper-
ature range examined.

Table II

Tensile Properties of MA 956E Sheet

Temp., (°F)	Longitudinal Direction			Transverse Direction		
	Tensile Strength		Elongation	Tensile Strength		Elongation
	(ksi)	(MPa)	(%)	(ksi)	(MPa)	(%)
75	104.4	(719.8)	13	103.8	(715.7)	14
1600	17.5	(120.7)	8	17.9	(123.4)	14
1800	15.3	(105.5)	8	15.2	(104.8)	7
2000	13.6	(93.8)	4	13.6	(93.8)	7
2100	12.2	(84.1)	2	8.4	(57.9)	3

In order to determine the stability of MA 956E to long-time exposure at
elevated temperature, specimens were exposed for 1000 hours at 1500°F
(816°C) with a stress of 4 ksi (27.6 MPa). Subsequent testing at room
temperature showed that no loss in strength or ductility occurred due to
the elevated temperature exposure.

Room Temperature Fabricability

One of the important properties of a sheet material intended for use in
gas turbines is its room temperature fabricability. The fabricability was
determined by measuring the bend angle of sheet specimens according to the
test method described in ASTM Specification E290. The angle for crack
initiation was recorded for a bend diameter equal to twice the sheet thickness
(nominally 0.05 inch).

Longitudinal and transverse specimens of MA 956E (bent about an axis
perpendicular or parallel to the rolling direction, respectively) consistently
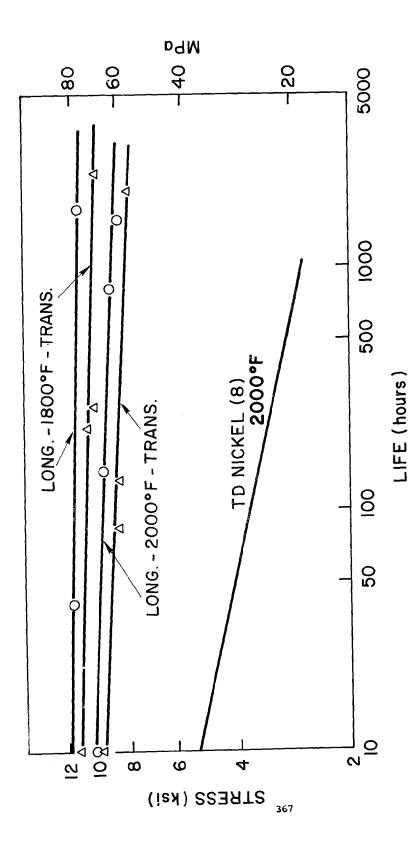achieved bend angles greater than 150° without cracking.

FIGURE 3 - STRESS RUPTURE CURVES AT 1800°F (980°C) AND 2000°F (1090°C) FOR MA 956E SHEET.

High Temperature Oxidation-Corrosion Resistance Cyclic Oxidation Tests
at 2012°F (1100°C)

It has been known for some time that ferritic iron-chromium alloys
containing more than about 4% aluminum possess exceptional resistance to
high temperature oxidation(10). The oxidation resistance is the result of
the formation of a thin adherent layer of Al_2O_3 on the surface of the metal
during the initial exposure period. Kornilov(11) found that for exposure
temperatures below about 700°C, the scale consists of a solid solution of
Al_2O_3, Fe_3O_4, and Cr_2O_3. As the temperature of exposure is increased above
700°C, the amount of Al_2O_3 present in the scale increases substantially and
for exposures above 1000°C the scale consists primarily of Al_2O_3.

The oxidation resistance of MA 956E was measured at 2012°F (1100°C) in
air-5%H_2O atmosphere at a flow rate of 250/cm^3/min. The samples were
cycled to room temperature every 24 hours in order to determine the change
in weight. A sample of TDNiCr was included in the test for reference
purposes. The results are given in Table III. After 504 hours the MA 956E
sample showed a small weight gain of 0.99 mg/cm^2 compared to a weight loss
of 4.6 mg/cm^2 for the TDNiCr specimen. X-ray diffraction scans of the
oxide scale of MA 956E revealed only Al_2O_3 was present after testing. The
descaled weight loss for the MA 956E sample was 1.57 mg/cm^2 (31×10^{-4}
mg/cm^2-hr) which is similar to a value of 35×10^{-4} mg/cm^2-hr obtained by
Kornilov(11) on a non-dispersion strengthened Fe-25Cr-5.6Al alloy tested at
1100°C. Note also, the exceptionally low values obtained by the metal loss
and maximum attack measurements.

Table III

1100°C (2012°F) Cyclic Oxidation Results

Test Time   : 504 Hours
Atmosphere   : Air + 5% H_2O at 250 cm^3/min
Thermal Cycle: To Room Temperature Every 24 Hours

Alloy	Weight Change (mg/cm^2) Undescaled	Descaled	Metal Loss[1] (µm)	Max. Attack[2] (µm)
MA 956E	0.99	− 1.57	<2	15
TDNiCr	−4.66	−12.52	20	33

[1]Metal loss measurement is defined as specimen thickness
before testing minus descaled specimen thickness after
testing.
[2]Maximum attack is defined as specimen thickness before
testing minus specimen thickness showing no evidence of
corrosion of oxidation attack after testing.

Burner-Rig Test at 1700°F

The resistance of the MA 956E alloy to hot corrosion attack by jet fuel
products was measured by testing in a burner-rig at 1700°F (927°C). The
apparatus used is similar to that described by Bergman et al(12). The fuel
mixture consisted of JP-5 fuel with additions of 0.3% sulfur and 5 ppm
seawater. The test cycle consisted of 58 minutes at temperature followed
by a two minute rapid cool to near room temperature. Measurements of the
weight change of the specimens were made every 24 hours.

The results obtained on MA 956E and TDNiCr are listed in Table IV along
with measurements of the amount of corrosion attack. The MA 956E specimen

showed a weight gain of only slightly more than 1 mg/cm^2 after the 312 hours of test time. Measurements of both the metal loss and depth of attack were less than 1 mil (25μm).

The structure of the exposed MA 956E alloy is shown in Figure 4. The sheet specimen showed some fine porosity near the surface but no evidence of sub-surface corrosion attack.

Table IV

1700°F Burner-Rig Results

Test Time   : 312 Hours
Atmosphere   : JP-5 Fuel + 0.3% Sulfur + 5 ppm Seawater
Thermal Cycle : 58 min. at Test Temperature Followed by
                Rapid Cool to Room Temperature in ∿2 min.
Air/Fuel Ratio: 30/1

Alloy	Weight Change (mg/cm^2) Undescaled	Descaled	Metal Loss (μm)	Max. Attack (μm)
MA 956E	1.04	– 0.17	5	18
TDNiCr	-1.69	-11.57	25	129

Carburization Test at 2000°F

In order to determine the carburization resistance of MA 956E, the alloy was exposed to a dynamic hydrogen-2% methane atmosphere for 100 hours at 2000°F (1093°C). A commercial Fe-Ni-Cr alloy was included in the test for comparison purposes. The results given in Table V show the weight gain values for MA 956E were well below 1 mg/cm^2 and the metal loss and depth of attack values were only about 0.1 mils (3μm). Electron microprobe analysis provided further evidence that carburization attack was minimal in that a carbon concentration of only approximately 0.03% was measured at the metal surface after exposure.

Table V

2000°F Carburization Test Results

Test Time : 100 Hours
Atmosphere: H$_2$ + 2%CH$_4$
Flow Rate : 1000 cm^3/min

Alloy	Weight Change (mg/cm^2) Undescaled	Descaled	Metal Loss (μm)	Max. Attack (μm)
MA 956E	0.51	0.16	3	3
INCOLOY* alloy 800	33.74	29.89	132	1620

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Summary

This investigation has shown that a ferritic Fe-Cr-Al alloy can be dispersion strengthened via the mechanical alloying technique to produce a sheet material, MA 956E, with excellent high temperature properties.

← Surface

500X

FIGURE 4

SURFACE STRUCTURE OF MA 956E SPECIMEN AFTER TESTING
IN BURNER RIG FOR 312 HOURS AT 1700°F

The microstructure of the alloy is characterized by large pancake-shaped grains with an aspect ratio of greater than ten in the plane of the sheet. In addition, electron microscopy revealed that the internal structure contains a uniform distribution of fine yttria-aluminate particles. It seems likely that the combination of these microstructural features is the major contributing factor in the alloy's isotropic, high temperature strength. The stress rupture results obtained on the MA 956E sheet material at 2000°F showed the alloy's properties are superior to those of TD nickel.

Fabricability tests on MA 956E sheet specimens consistently achieved bend angles greater than 150° about a diameter equal to twice the sheet thickness.

The cyclic oxidation, burner-rig and carburization tests on MA 956E show the alloy is extremely resistant to elevated temperature attack. In all of these tests MA 956E showed only a slight weight gain after the long time exposures. The small weight gain is undoubtedly due to the formation of an adherent Al_2O_3 coating on the metal surface. Microstructural examination of the specimen tested in the burner-rig revealed there was no evidence of subsurface corrosion attack.

The results obtained in this study, therefore, indicate the MA 956E alloy possesses an excellent combination of high temperature strength, fabricability and oxidation-corrosion resistance. These properties, together with the high melting point, low density and low thermal expansion coefficient that are characteristic of a ferritic composition suggest the alloy would have good potential for use in a variety of high temperature sheet applications.

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