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Incoloy 908 Database Report
On Process - Structure - Property Relationship

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ABSTRACT

Incoloy 908 is a nickel-iron base superalloy with a coefficient of expansion (COE) and mechanical properties that have been optimized for use in Nb_3Sn superconducting magnets. It has been proposed for use as a conduit material for the International Thermonuclear Experimental Reactor (ITER) magnets. The relationship between manufacturing processes, microstructures and mechanical properties of Incoloy 908 are characterized in support of the magnet fabrication and quality control. This report presents microhardness, microstructure, and yield and ultimate tensile strengths as functions of thermomechanical process variables including heat treatment, annealing and cold work for laboratory prepared Incoloy 908 specimens. Empirical correlations have been developed for the microhardness at room temperature and tensile strength at room temperature and at 4K. These results may be used for manufacturing quality control or for design.

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Illustrations and Tables:

Introduction:

Incoloy® alloy 908 is a nickel-based superalloy developed for use in Nb_3Sn cable-in-conduit-conductor (CICC) magnets. The alloy has thermal coefficient of expansion (COE) properties which closely match those of Nb_3Sn, minimizing compressive strain in the superconductor and the associated reduction in critical properties (upper critical field, critical temperature and critical current). Incoloy 908 also has mechanical properties at cryogenic temperatures which compare favorably with other metals.

Purpose:

The purpose of this report is to establish a process-structure-property relationship for Incoloy 908. This has been done by examining data on the microhardness and microstructural characteristics as a function of thermomechanical processes, including cold work and heat treatment and to relate these properties to the more useful tensile characteristics of the alloy.

The information in this database may be useful as a reference to compare microstructures. One can estimate tensile properties σ_y and σ_{UTS} at different temperatures (4.2K and 298K) based on microstructure and the processing history or by means of a simple, nondestructive Vickers or Rockwell C hardness test of the material in question.

It is also possible to take microhardness and grain size measurements and determine with a high level of confidence whether the material was annealed, heat treated, cold worked or as-received. This practice can be used for quality control purposes in the conduit/magnet fabrication processes.

Also of interest prior to this compilation was the effect of annealing on cold worked Incoloy 908. It is now apparent that solution annealing at 1050°C for one hour reverses the effects of cold work completely, while the mill anneal at 980°C for one hour was not as effective.

Summary:

1. Tensile properties including yield strength (σ_y, defined at 0.2% offset), and ultimate tensile strength (σ_{UTS}) were found to be related to Vickers microhardness as shown in Figures 1 and 2 below.

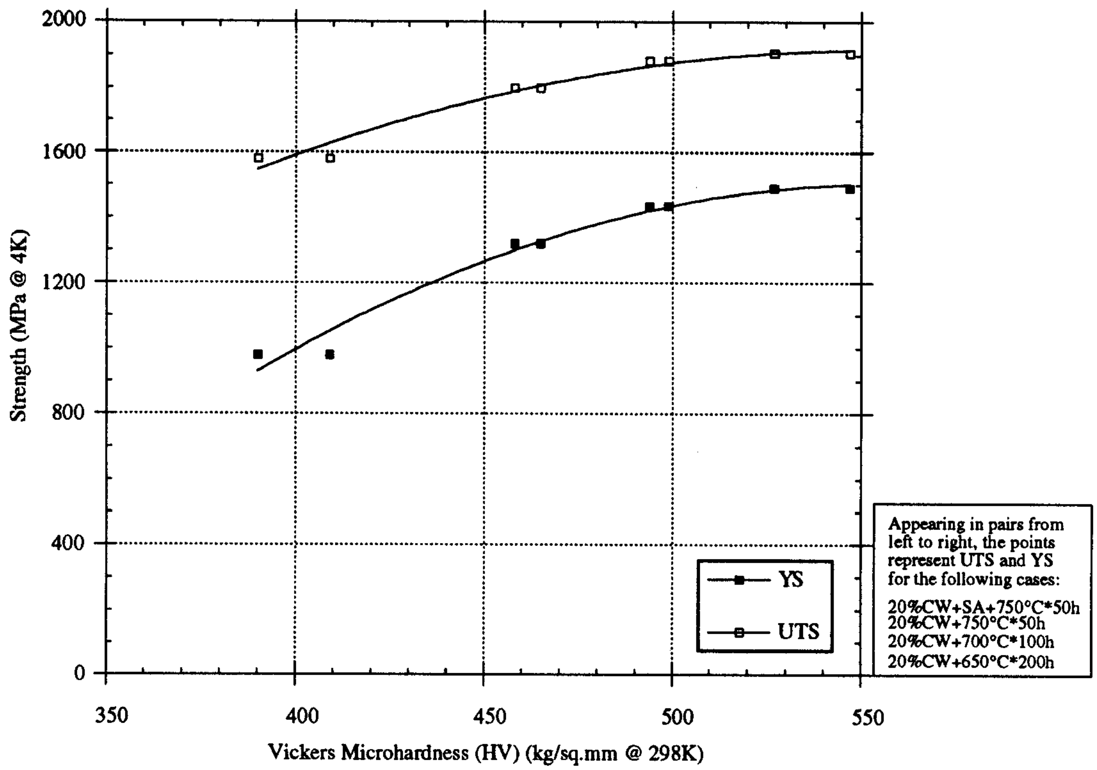


Figure 1. Yield (0.2% offset) and ultimate tensile strength of Incoloy 908 at 4K plotted against Vickers microhardness at 298K. Cold work (CW), solution anneal (SA) and vacuum heat treatment at the specified temperatures and times are the given parameters. (6)

A second order polynomial curve fit for the data in Figure 1 generated the following two equations for yield strength and ultimate tensile strength at 4 K (in MPa) as functions of Vickers microhardness (kg/sq.mm) at room temperature:

$$\sigma_{YS@4K}(HV) = -4782.02 + 22.4901 * (HV) - 0.0201146 * (HV)^2$$

$$\sigma_{UTS@4K}(HV) = -2363.18 + 15.48898 * (HV) - 0.0140185 * (HV)^2$$

For conversion purposes, Vickers microhardness ≈ Rockwell C hardness + 10 (see Table 4, page 19 for more exact conversion) and 1 MPa ≈ 145 psi.

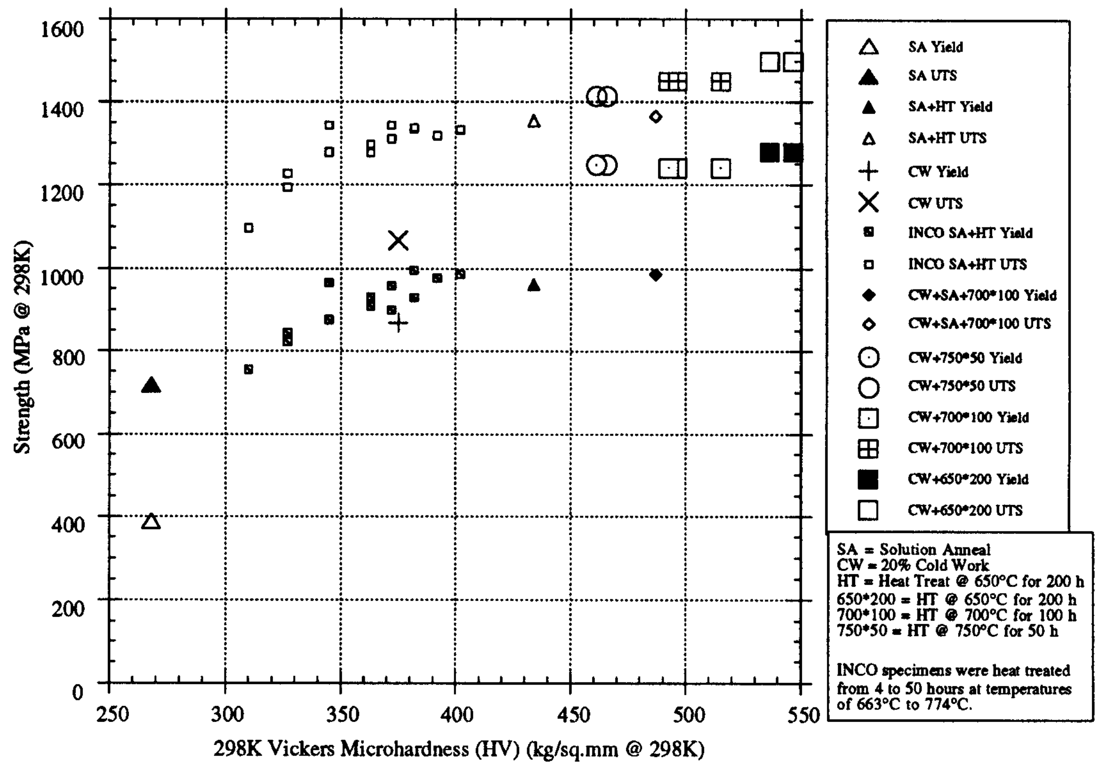


Figure 2. Yield (0.2% offset) and ultimate tensile strength of Incoloy 908 at 298K plotted against Vickers microhardness at 298K. Yield and ultimate tensile strengths for each case observed are shown in pairs with similar icons (each pair has the same microhardness value). All UTS values are higher than YS. [6,15]

2. Both YS and UTS of the annealed alloy are increased by up to 750 MPa when it is cold worked and/or aged and tested at room temperature. Both processes combined produce the best tensile strengths (up to σ_y = 1489 MPa and σ_{UTS} = 1906 MPa at 4.2K for samples cold worked 20% and heat treated at 650°C for 200 hours). The lowest tensile properties (σ_y= 389 MPa , σ_{UTS} = 717 MPa) were found by testing the alloy as received (mill annealed) at room temperature. [6]

3. Some plates of thickness 1/4" or greater have a few "elephant" grains scattered throughout them. These grains are much larger than the normal surrounding grains, and are oriented in thin, elongated disks in planes parallel to the plane of the plate. These appear to be examples of incomplete recrystallization, possibly similar to those observed in alloy 718.[17] Some of these elephant grains have faint internal grain boundaries, while others do not. These grains have been named Type I and Type II, respectively, for convenience. Figure 3 shows one of each at 200x, and Figure 4 shows part of one Type II elephant grain at 500x.

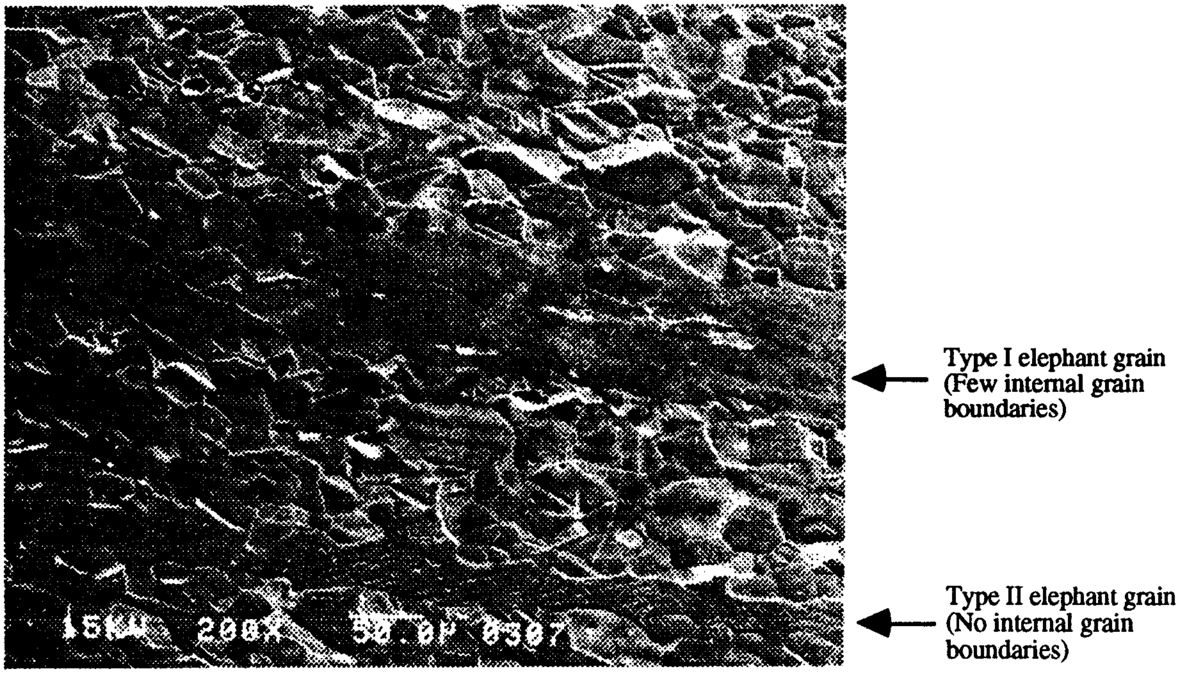


Figure 3. Type I elephant grain runs across the middle of this photomicrograph; note that faint internal grain boundaries may be seen at the left hand side of this grain. A type II elephant grain (with no internal grain boundaries) runs across the bottom. See Figure 4 below for an enlargement. (Photomicrograph taken on a scanning electron microscope (SEM) at 200x; 10% cold worked specimen, long transverse orientation).

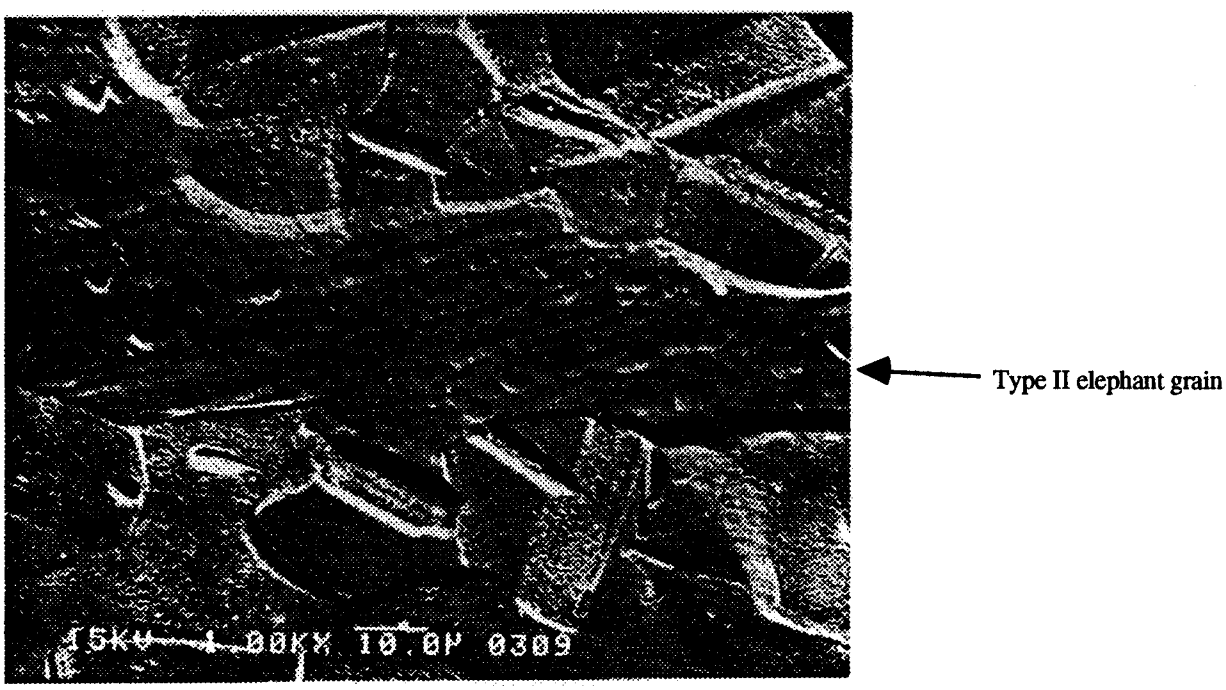


Figure 4. Type II elephant grain @1000x on SEM (same specimen as above)

These elephant grains were measured to be about 4% harder than normal grains. Energy dispersive x-ray chemical analysis suggests that the elephant grains have about 14% more chromium and titanium than other grains (see Table 5). However, they are *completely absent* from both solution annealed plate and extruded Incoloy 908, and they have not been found in plates less than 1/4" thick.

4. The carbide particles, (Nb,Ti)C appear to be less common in aged specimens than in annealed ones. This may result in a more homogeneous chemistry and possibly better properties in the aged alloy. In both cases the particles typically measured about 5.5 microns. Larger particles ranged up to about 10 by 25 microns, but these were less common.

5. Solution annealing Incoloy 908 at 1050°C for one hour removes the effects of cold work but substantially increases grain size. It likely has the same effect on aged Incoloy 908. The alloy may be worked and annealed multiple times, then aged to achieve the desired form and properties. INCO has demonstrated this very clearly in the manufacture of round, seamless extruded tubing for use in CICC magnets. It is not known how grain size and stress-accelerated grain boundary oxidation (SAGBO) properties of Incoloy 908 are related.

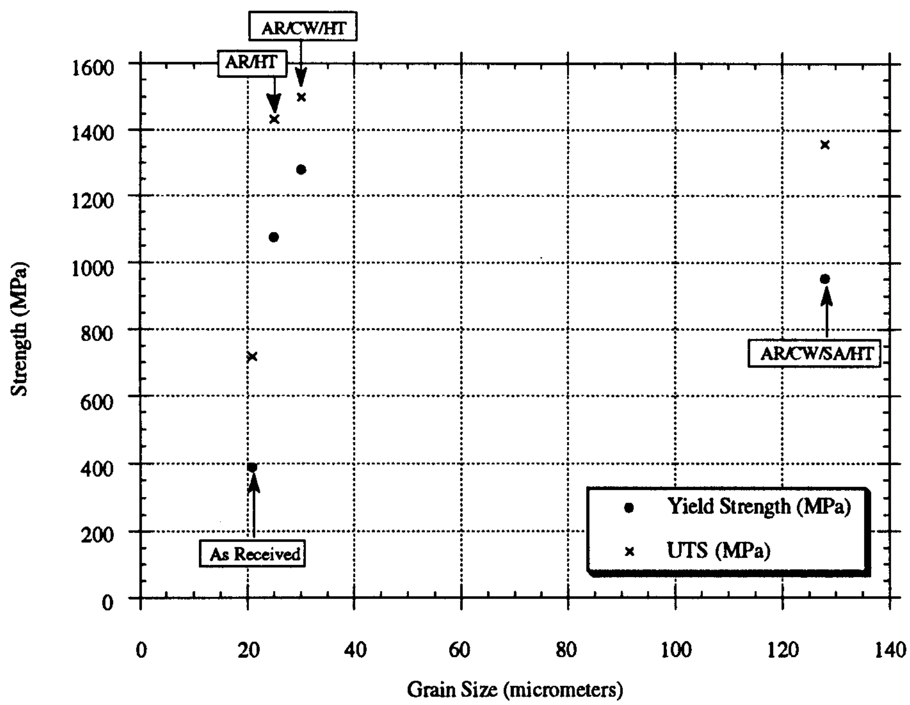


Figure 5. Yield and ultimate tensile strength (@ 298K) plotted against grain size. [6]

6. Figure 5 shows that yield and ultimate tensile strength do not appear to be strongly related to grain size.

7. The mill annealed alloy is nearly isotropic. It is approximately 5% harder along the transverse orientations compared to the longitudinal sections. Tables 6a, b and c in Appendix B show that the differences in microhardness among the three orthogonal planes are very small. There were no discernible trends among the data except that solution annealing made the alloy almost perfectly isotropic.

8. INCO has produced Incoloy 908 to specifications. Properties of the two heats, Y 9210 K and Y 9400 K were very similar. Table 3 in Appendix B shows the chemistry of the two heats to be consistent, and Tables 6a, b ,c and 7 in Appendix B show that the two heats had similar microstructure and microhardness.

Material Characterization Procedure:

1. Sample Preparation:

Incoloy 908 was produced by vacuum induction melting by INCO Alloys International, Huntington, WV. The ingots were homogenized at 1191°C for 16 hours and fast cooled. A series of forging and reheating steps produced plates that were mill annealed at 980°C for one hour and air cooled. [6]

Test specimens were prepared from two heats; Y 9210 K (August 1987) and Y 9400 K (May 1992).

<u>Heat Y 9400 K</u>

Heat Y 9400 K (1992) was received in plates measuring 6 feet by 6 inches by 0.252 inches. One of these plates was cut into six pieces each measuring 12 inches by 6 inches by 0.252 inches. The first piece was kept as-received. The second was passed through a cold rolling mill about thirty times, reducing its cross-sectional area from 6.141"x0.252" to 6.208"x0.227". Cold work is defined as the percent reduction in cross-sectional area: the second piece was cold worked

$$1-\frac{(6.208*0.227)}{(6.141*0.202)}=0.091=9.1\%$$

and the sixth piece was cold rolled from 6.141"x0.252" to 6.288"x0.202" for a total reduction of 18.0% in about ninety passes. Pieces 3, 4 and 5 were not used.

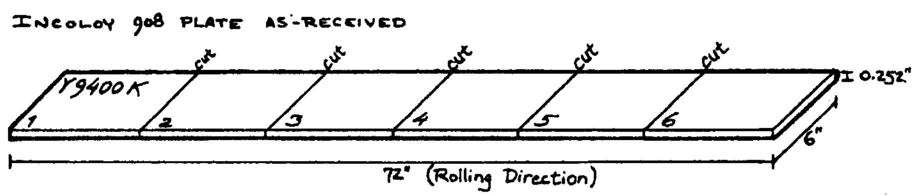


Figure 6. 1/4" Incoloy 908 plate as received, showing cuts made prior to the 9% and 18% cold working of sections 2 and 6.

Twelve samples sized approximately 1/4"x3/8"x1/2" were cut from each of the three plates (36 samples total) with a low speed diamond saw as shown in Figure 6. These dimensions were chosen to assure that the orientations of the samples were easily identifiable.

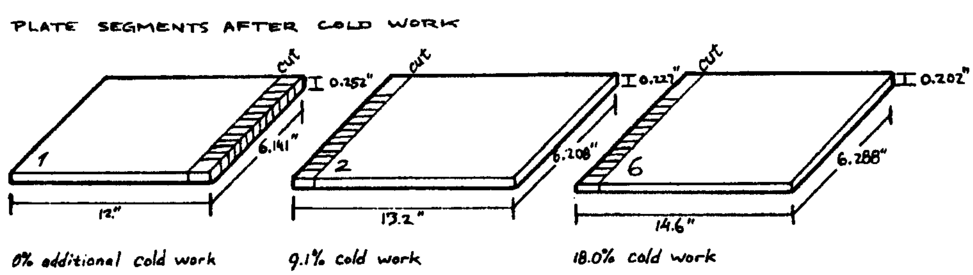


Figure 7. Segments of Incoloy 908 plate after cold worked 0, 9 and 18%.

Three samples from each plate were mounted as cut, with one face representing each of the three orientations: Longitudinal (L), Short Transverse (S), and Long Transverse (T), and three mounts representing each of the three states of cold work (0%, 9.1% and 18.0%) were prepared.

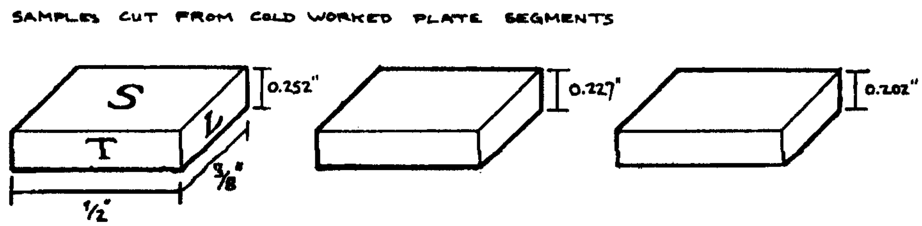


Figure 8. Individual samples of Incoloy 908 cold worked 0, 9 and 18%, showing orientation and dimensions.

All three orientations are represented in each mounted sample as shown in Figure 8 below. By observing the differences in properties among the three orientations, one can estimate how processing affects the anisotropy of the metal.

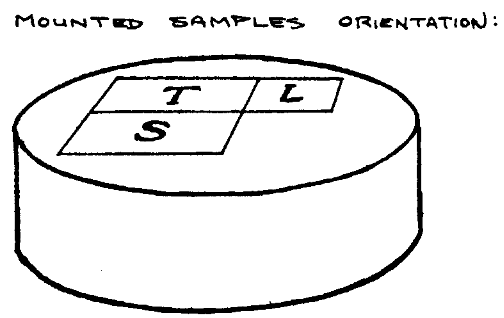


Figure 9. Standard orientation of samples in each mounted specimen. All three samples in each mounted specimen were cold worked a given amount and heat treated under the same conditions.

<u>Cold Worked (0, 9 & 18%)</u>

Three samples from each plate were mounted, representing the as-received and as-received plus cold worked conditions.

<u>Cold Worked (0, 9 & 18%) and Solution Annealed</u>

Three samples from each plate were solution annealed in air at 1050°C for one hour, then water quenched to 24°C, cleaned and mounted.

<u>Cold Worked (0, 9 & 18%) and Heat Treated</u>

Three samples from each plate were heat treated at 650°C for 200 hours in vacuum to simulate the heat treatment conditions of the conduit during the Nb_3Sn formation. The samples were then cleaned and mounted.

<u>Cold Worked (0, 9 & 18%), Solution Annealed and Heat Treated</u>

The remaining nine samples were solution annealed in air at 1050°C for one hour, water quenched and then heat treated in vacuum at 650°C for 200 hours to determine how well the annealing process would reverse the effects of the cold work and what effect the anneal would have on the final properties of the alloy. These three samples were then cleaned and mounted.

Heat Y 9210 K

Cold Worked 0% (none), Flash Welded and Heat Treated

Three samples representing heat Y 9210 K (1987) were cut from the base metal of a tensile test specimen that had been flash welded and heat treated. In flash welding, two pieces of metal are faced together and heated by passing a large electrical current through them. Then the work pieces are forged to make a bond. Flash welding does not form a dendritic weld zone and it has little or no effect on the microstructure of the base metal outside of the weld zone, so it is potentially stronger than more conventional welds. The samples were cleaned, mounted and then used to compare the 1987 and 1992 heats.

All mounted samples were ground with silicon carbide paper as fine as 500 grit, and then polished with 0.05 mm alumina powder. Each was then etched with a three acid stainless steel etchant (10 ml nitric acid, 10 ml acetic acid, 15 ml hydrochloric acid, 6 drops glycerol) to reveal the grain boundaries. The different specimens were etched for different times to produce the desired relief. Etching times varied from about fifteen seconds for the aged specimens to about forty-five seconds for the solution annealed ones. It was noted that the aged specimens stained very easily, which may be caused by the evolution of the γ' phase within the matrix.

2. Grain Size Measurements:

The etched specimens were photographed under Carl Zeiss optical microscope (serial #4312391) with Polaroid Land Camera film Types 52 or 55. Two photographs were taken of each condition and each orientation at magnifications of 50x or 100x to provide grain boundary intersects as specified by ASTM Method E112 for the measurement of grain size by the linear intercept method. The diagonals of each picture were chosen as clearly defined lines of uniform length along which the grain boundary intercepts could be counted. The four intercept counts as shown below were averaged for each case. This average was then divided by the true length of the photo diagonals, which were 1.45 mm for 100x photos and 2.90 mm in the 50x photos. The values for ASTM grain size G, nominal grain diameter d_n and Feret's grain diameter d_f were interpolated from ASTM Method E112 Table 2 using the following equation [2]:

$$G, d_n, \text{ or } d_f = \frac{(B-C)}{(A-C)} * (D-E) + E$$

A = Next lower intercept count on table
B = Intercept count
C = Next higher intercept count on table
D = G, d_n or d_f corresponding to A
E = G, d_n or d_f corresponding to C

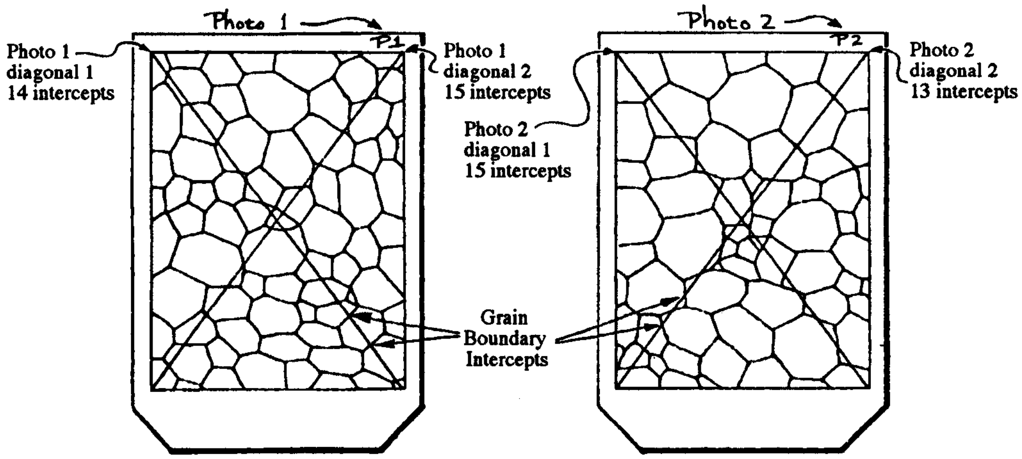


Figure 10. Grain size picture illustrating intercept count technique.

Table 1. Values corresponding to the intercept counts for the sample illustrated in Figure 10, taken from ASTM section E112 table 4: "Macro-Grain Size Relations Computed for Uniform Randomly Oriented Equiaxed Grains" (2)

Intercept Count *(intercepts/mm)*	ASTM Grain Size "G"	Nominal Diameter d_n *(mm)*	Feret's Diameter d_f *(mm)*
A = 4.42	D_1 = 1.0	D_2 = 25	D_3 = 28.5
C = 5.26	E_1 = 1.5	E_2 = 21	E_3 = 24.0

Table 2. Interpolated G, nominal and Feret's grain size for the sample illustrated in Fig. 10.

Magnification:	**50x**
Photo P1: 1st, 2nd diagonal intercepts	**14** **15**
Photo P2: 1st, 2nd diagonal intercepts	**13** **15**
Average number of intercepts	$\frac{14+15+15+13}{4} = \mathbf{14.25}$
Intercept Count *(intercepts/mm)*	$\frac{14.25}{2.90} = \mathbf{4.914}$
ASTM "G"	$\frac{(4.914-5.26)*(1.0-1.5)}{4.42-5.26} + 1.5 \approx \mathbf{1.5}$
Nominal d_n *(mm)*	$\frac{(4.914-5.26)*(25-21)}{4.42-5.26} + 21 = \mathbf{22.6}$
Feret's d_f *(mm)*	$\frac{(4.914-5.26)*(28.5-24)}{4.42-5.26} + 24 = \mathbf{25.9}$

3. Vickers (diamond) microhardness measurement:

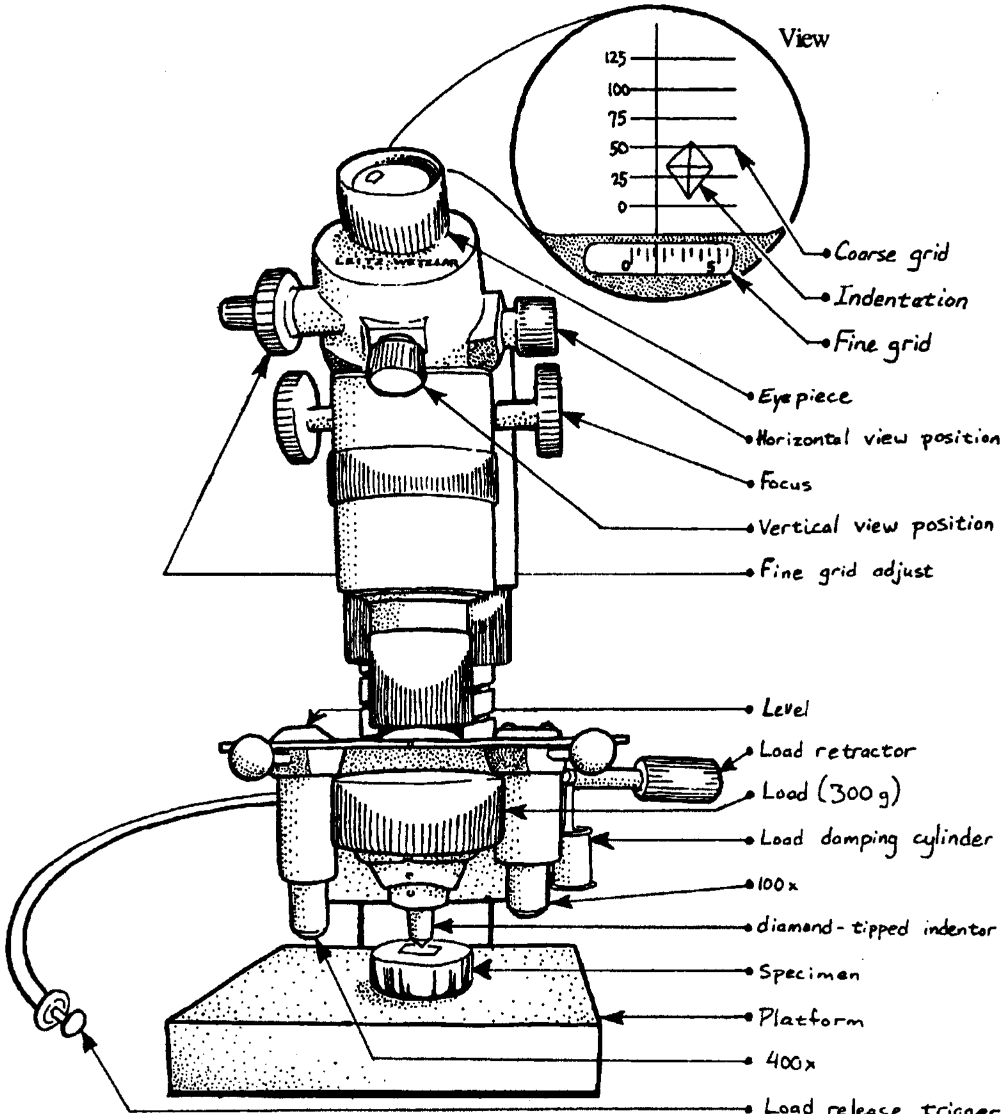


Figure 11. Leitz-Wetzlar microhardness tester.

All microhardness measurements were performed on a Leitz-Wetzlar microhardness tester using a 300 gram indentor weight. The weight, when released, slowly drives an oil damped mechanism which presses a diamond indentor into the specimen placed on the platform. The indentor stops when it has pressed deeply enough into the specimen to counteract the force applied by the weight, typically within 15 to 30 seconds. When the weight is removed, a square indent is left behind in the surface of the test specimen. The length of the square's vertical diagonal (height as seen through the eyepiece at 400x) is measured by aligning the 0.5 μm grid with the corners of the indent. The horizontal diagonal (width) is measured by rotating the upper portion of the microscope 90° and repeating the procedure. Ten indent tests were taken for each condition and each orientation and the diagonal sizes were averaged for each case. Vickers microhardness is calculated with the following formula:

$$HV = \frac{1854*P}{d^2} \quad kg/mm^2$$

where P = indentor weight in grams
and d = average indent diagonal length in micrometers

See Table 4 in Appendix B for approximate Vickers to Rockwell C conversions. In general, Vickers microhardness is a factor of ten greater than Rockwell C.

4. Tensile Property Measurement:

Yield and Ultimate Tensile Strength were measured in accordance with ASTM Method E8. Detailed procedures are described in "Mechanical Properties of Incoloy 908, an Update". [4]

Discussion:

Hardness versus cold work

As-received Incoloy 908 has been hot rolled and mill annealed at 980°C for about an hour prior to delivery. It has a Vickers microhardness (HV) of about 268 kg/mm^2 and tensile properties σ_y = 389 and σ_{UTS} = 717 MPa [6]. Vickers microhardness measurements taken from as-received material suggest that when Incoloy 908 is cold worked by 9.1%, the Vickers microhardness of the as-received alloy increases by about 84 kg/sq. mm (31%); 18% cold work produces an microhardness increase of 107 kg/sq. mm (40%). INCO Alloys laboratory results taken from solution annealed and cold worked samples show a similar pattern, with hardness increasing by up to 187 kg/sq. mm above the initial value.

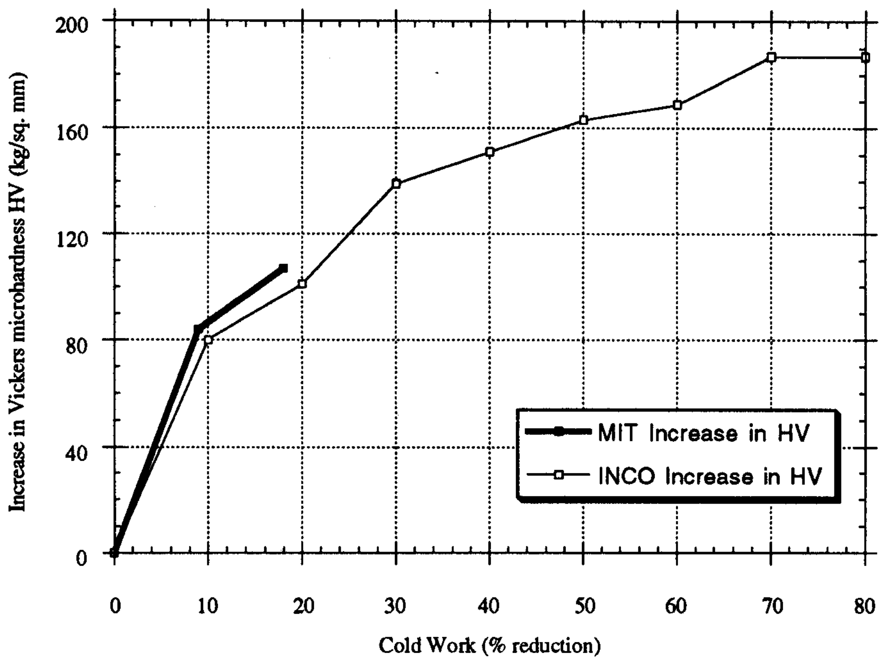


Figure 12. Increase in hardness as a function of percentage cold work [15]

The discrepancy between the two profiles is due to the use of two different procedures and test methods. The INCO specimens were annealed and tested using Rockwell A or superficial hardness tests (converted using ASTM section E 140 Table 3, "Approximate Hardness Conversion Numbers for Nickel and High-Nickel Alloys")[1], while the MIT specimens were cold worked as-received and tested with Vickers microhardness equipment. Nevertheless, the two curves corroborate each other, and they show that the alloy's highest tensile properties can be approached most easily by cold working the metal 30%.

This curve should be useful for conduit design purposes, because it allows one to predict the hardness and corresponding tensile properties of cold worked Incoloy 908.

Elephant grains

Thick rolled plate may contain elephant grains that appear to be examples of incomplete recrystallization, a phenomenon that has been observed in alloy 718.[17] Some of these grains appear to be more resistant to etching as they show very faint grain boundaries, while others show no evidence of internal grain boundaries even when etched very heavily. These elephant grains tend to be about 4% harder than the surrounding normal grains (and likely have higher tensile properties to match), and fatigue fracture surfaces have been found to propagate along them. X-ray spectroscopy performed under the scanning electron microscope (SEM) using Noran Voyager System Software suggests that chromium and titanium levels in the elephant grains may be elevated about 14% above those of normal grains (4.52 wt% Cr vs. 3.98 wt% and 1.70 wt% Ti vs. 1.48 wt%). Please see Table 5 (Appendix B) for more details. Error in these measurements may be as high as 0.5 wt%, but the results showed consistently higher chromium and titanium content in elephant grains. However, it should be noted that this phenomenon in Incoloy 908 has been found only in plate of 1/4" or greater thickness; no such elephant grains were observed in extruded Incoloy 908 prepared by Climax and INCO.

Carbide density

Carbide number density appears to be about 85% higher in the as-received and solution annealed samples compared to the aged ones. Microhardness tests on carbide "stringers", or lines of carbides, suggest that hardness values are about 4% higher than those taken in the surrounding, carbide-free grains.

Effect of Annealing

This research has established that solution annealing at 1050°C for one hour will reverse the effects of cold work, while the 980°C mill anneal is not as effective.

Appendix A: Glossary

- **Miscellaneous:**

CICC: Cable-In-Conduit-Conductor: Superconducting cable is sheathed in a tube which provides structural support for the cable and contains the liquid helium coolant.

HA/IAII: Huntington Alloys/INCO Alloys International, Inc.
Post Office Box 1958
Huntington, West Virginia, USA 25720

SAGBO: Stress-Accelerated Grain Boundary Oxidation. Some nickel based superalloys have a tendency to undergo oxidation at the grain boundaries when exposed to oxygen and surface stress exceeds a given percentage of yield strength. This may lead to intergranular cracking. The SAGBO threshold of Incoloy 908 is currently being explored.

- **Processes:**

Age (HT): Precipitation hardening of the alloy by heat treating at 650°C for 200 hours in vacuum. These are the conditions under which the Nb_3Sn reaction takes place in the manufacture of superconducting magnets.

As-received (AR): Hot rolled and mill annealed at 980°C for one hour

Cold Work (CW): Percentage reduction of cross-sectional area; all cold work in this report was performed on a rolling mill. Cold working metals creates dislocations in the crystalline structure of the matrix. These dislocations cause the metal to harden in a process known as strain hardening. There is a corresponding increase in the tensile strength of the metal.

Flash Weld (FW): A welding process in which two pieces of metal are faced together while a strong electrical current is passed through them. The pieces are then pushed together, and are joined by a very fine weld line instead of the more conventional weld zone. Flow patterns may be visible in the metal immediately surrounding the weld line due to the displacement of the base metal during the welding process.

Hardness: A measure of the resistance of a metal to plastic deformation. (16)

Mill Anneal: Annealing of Incoloy 908 at 980°C for one hour by INCO Alloys.

Solution Anneal (SA): Annealing of Incoloy 908 at 1050°C for one hour to remove strain hardening. The annealing process causes recovery, recrystallization and grain growth, creating a supersaturated solid solution.

- **Orientation:**

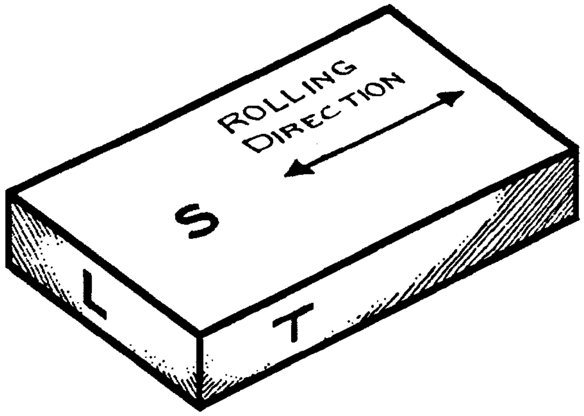


Figure 13. Illustration showing the three orientations and the rolling direction.

Longitudinal orientation (L): Plane perpendicular to the rolling direction

Short Transverse (Normal) orientation (S): This section is coplanar to the two largest surfaces of the rolled plate.

Long Transverse orientation (T): Plane perpendicular to both Longitudinal and Short Transverse sections.

- **Tests:**

Hardness measurements:

Vickers Microhardness (HV or VHN): Vickers microhardness is measured by pressing a tetrahedral diamond-tipped indentor into the specimen with a given load. Microhardness is a function of applied load and the width of the indentation. (2)

$$HV = \frac{1854*P}{d^2}$$

where P = indentor weight in grams
d = average length of indent diagonals in microns
$\alpha = 136°$ angle between opposing faces of the diamond indentor
Microhardness typically can be related to tensile properties of metals by a second order polynomial equation.

Rockwell C Hardness (HRC): Rockwell hardness is a function of load and indentation depth. Rockwell A,C and D tests use a diamond cone indentor, while Rockwell B,F,G and E tests use a hardened steel ball indentor. (7)

Grain Size measurements:

Feret's Grain Diameter (d_f): Height between tangents (Average grain section area / Average intercept distance)

Grain Boundary Intercepts: Intersections of the test pattern with grain boundaries.

Grain Size: The area within the confines of the original (primary) boundary.

Intercept Count: The number of Grain Boundary Intercepts divided by the length, in millimeters, of the sample.

Nominal Grain Diameter (d_n): Geometric mean of the intercept width and Feret's diameter (not directly measurable) (2)

Tensile test properties:

UTS (Ultimate Tensile Strength): As a tensile specimen is pulled in the plastic (nonlinear) regime, its cross-sectional area tends to decrease or neck. Because the metal is undergoing work hardening, it can still support the increasing load. As a result of the increased load and the decreased cross-sectional area, stress in the specimen rises, until necking reduces the area faster than strain hardening increases the strength (causing failure). The ultimate tensile strength is the maximum stress that the metal can support in tension. See Figure 14.

YS (Yield Strength): Stress at 0.2% plastic strain. Yield strength approximates the transition point between elastic (linear) and plastic (nonlinear) deformation. See Figure 14.

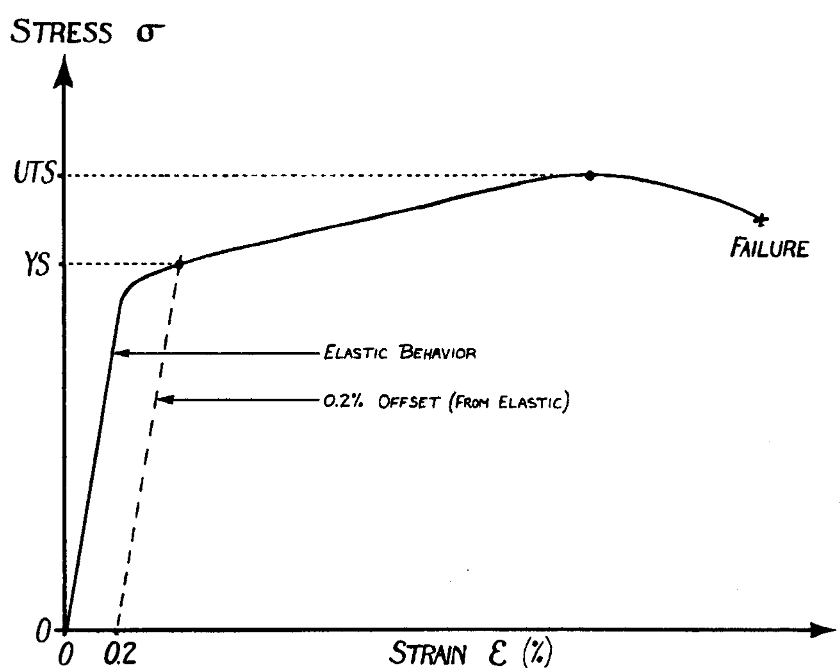


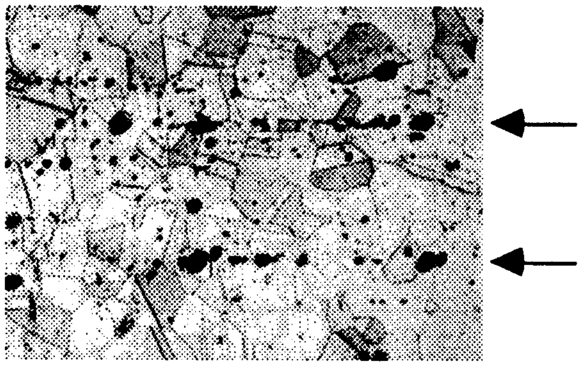
Figure 14. Typical Stress-Strain curve for a ductile metal, showing Yield and Ultimate Tensile Strength.

- **Incoloy 908 Characteristics:**

  γ' phase: Chemistry = $Ni_3(Nb,Al,Ti)$, an ordered intermetallic fcc phase; γ' is the primary strengthening phase in Incoloy 908 [11]

  Carbides: Chemistry = (Nb,Ti)C

  Carbide Stringers: Carbide stringers are arrays of carbides which were broken down from a larger carbide particle during the forging and cold work processes.



Arrows show carbide stringers that run across the photo.

Figure 15. Carbide stringers seen at 50x. Stringers are oriented in the same direction as the rolling direction of the Incoloy 908 plate, so they are visible in both transverse sections, but appear as individual carbide particles in longitudinal sections. Here they appear in a horizontal line across the middle of the photograph where the etchant dissolved the matrix around the carbides, leaving them at the centers of the dark pits.

Appendix B: Tables

Table 3. 1/4" Incoloy 908 plate chemistry [4,5]

Heat	7 mm plate Heat Y9210K	7 mm plate Heat Y9400K
Date	August 1987	May 1992
Processing	Hot rolled & mill annealed	Hot rolled & mill annealed
Analyst	HA/IAII	HA/IAII
Element		
Iron	40.96 wt%	40.77 wt%
Chromium	3.86	3.99
Cobalt	---	---
Manganese	0.04	0.05
Silicon	0.13	0.17
Niobium	2.99	3.02
Aluminum	0.97	0.98
Titanium	1.57	1.57
Carbon	0.01	0.01
Oxygen	---	---
Nitrogen	---	---
Phosphorus	---	0.005
Sulfur	0.001	<0.001
Boron	---	0.003
Nickel	49.46	49.42
Copper	0.01	0.01

Table 4. Approximate hardness conversion numbers. This conversion chart was estimated for nonaustenitic steels, but it is a relevant guideline for conversion of Incoloy 908 hardness. [10] See ASTM E 140 for further conversion charts. [1]

Vickers Hardness Number (HV)	**Rockwell C Hardness Number (HRC)**	**Vickers Hardness Number (HV)**	**Rockwell C Hardness Number (HRC)**	**Vickers Hardness Number (HV)**	**Rockwell C Hardness Number (HRC)**
560	53	402	41	294	29
544	52	392	40	286	28
528	51	382	39	279	27
513	50	372	38	272	26
498	49	363	37	266	25
484	48	354	36	260	24
471	47	345	35	254	23
458	46	336	34	248	22
446	45	327	33	243	21
434	44	318	32	238	20
423	43	310	31		
412	42	302	30		

Table 5. Results of Chemistry tests on Voyager Noran Energy Dispersive X-Ray Chemistry analysis system.

Aluminum, niobium, titanium, chromium, iron and nickel were analyzed by wt %. See Legend for explanation of terms.

	Al	Nb	Ti	Cr	Fe	Ni
Normal	0.82	3.86	1.28	4.57	41.52	47.95
	0.74	4.15	1.87	3.65	41.38	48.21
	0.79	3.32	1.30	3.71	41.88	48.99
Avg:	**0.783**	**3.777**	**1.483**	**3.977**	**41.593**	**48.383**
Diff:	*-20%*	*+25%*	*-6%*	*0%*	*+2%*	*-2%*
Type I	**0.94**	**3.68**	**1.70**	**4.56**	**41.97**	**47.14**
Diff:	*-4%*	*+21%*	*+8%*	*+14%*	*+3%*	*-4%*
Type II		4.17	1.49	4.35	42.58	47.41
	1.00	4.65	1.61	4.66	41.71	46.36
	0.85	3.71	1.65	4.65	42.35	46.79
	0.85	3.93	2.13	4.61	41.16	47.32
	0.82	3.11	1.45	4.14	42.86	47.61
	0.88	3.48	1.81	4.42	42.10	47.31
Avg:	**0.733**	**3.842**	**1.690**	**4.472**	**42.127**	**47.133**
Diff:	*-25%*	*+27%*	*+8%*	*+12%*	*+3%*	*-5%*
Expected	**0.98**	**3.02**	**1.57**	**3.99**	**40.77**	**49.42**

Legend:
Normal indicates a "typical" normal sized grain. Three samples taken as reference.
Type I indicates Type I elephant grain. Only one sample taken.
Type II indicates Type II elephant grain (no internal boundaries). Six samples taken.
Avg: is the average of any samples taken.
Diff: is the difference, in percent from the expected value.
Expected represents the chemical analysis supplied by INCO Alloys (See Table 3).

The estimated error for the x-ray chemistry analysis system is approximately 0.5 wt%. Most of the results presented here fall within that range. However, the difference in titanium and chromium contents between the elephant grains and the normal grains is fairly consistent and predictable. While it cannot be stated conclusively that there is a substantial difference in chemistry between the elephant grains and the normal ones based only on these limited tests, it may be a concern if heavy plates of Incoloy 908 are to be used for high fatigue applications.

Once again, these elephant grains seem to appear only in plate of 1/4" or greater, and can be eliminated by solution annealing or by extrusion.

Table 6a. Incoloy 908 1/4" plate - Longitudinal grain size and microhardness

Condition:	**1987 heat*** memo date	**Y9210K** nominal grain size *(micrometers)*	HV *(kg/sq.mm)*	**1992 heat** memo date	**Y9400K** nominal grain size *(micrometers)*	HV *(kg/sq.mm)*
AR	-	-	-	8/7/1992	21	263
AR+9%	-	-	-	8/7/1992	20	334
AR+18%	-	-	-	8/7/1992	21	362
AR+SA	-	-	-	8/14/1992	105	161
AR+9%+SA	-	-	-	8/14/1992	99	172
AR+18%+SA	-	-	-	8/14/1992	106	163
AR+Age	7/6/1992	25	503	8/28/1992	25	481
AR+9%+Age	-	-	-	8/28/1992	25	483
AR+18%+Age	-	-	-	8/28/1992	28	502
AR+SA+Age	-	-	-	9/16/1992	119	407
AR+9%+SA+Age	-	-	-	9/16/1992	111	433
AR+18%+SA+Age	-	-	-	9/16/1992	117	422

Table 6b. Incoloy 908 1/4" plate - Short Transverse grain size and microhardness

Condition:	**1987 heat*** memo date	**Y9210K** nominal grain size *(micrometers)*	HV *(kg/sq.mm)*	**1992 heat** memo date	**Y9400K** nominal grain size *(micrometers)*	HV *(kg/sq.mm)*
AR	-	-	-	8/7/1992	20	270
AR+9%	-	-	-	8/7/1992	21	359
AR+18%	-	-	-	8/7/1992	25	357
AR+SA	-	-	-	8/14/1992	88	163
AR+9%+SA	-	-	-	8/14/1992	110	169
AR+18%+SA	-	-	-	8/14/1992	105	166
AR+Age	7/6/1992	31	502	8/28/1992	24	485
AR+9%+Age	-	-	-	8/28/1992	27	489
AR+18%+Age	-	-	-	8/28/1992	34	508
AR+SA+Age	-	-	-	9/16/1992	79	436
AR+9%+SA+Age	-	-	-	9/16/1992	87	485
AR+18%+SA+Age	-	-	-	9/16/1992	134	424

Legend:
AR = As Received
9% = 9% Cold Worked
18% = 18% Cold Worked
SA = Solution Annealed at 1050°C for one hour
Age = Heat Treated at 650°C for 200 hours
* 1987 specimen was flash welded
Vickers microhardness measured with a 300 gram load.

Table 6c. Incoloy 908 1/4" plate - Long Transverse grain size and microhardness

Condition:	1987 heat* memo date	Y9210K nominal grain size *(micrometers)*	HV *(kg/sq.mm)*	1992 heat memo date	Y9400K nominal grain size *(micrometers)*	HV *(kg/sq.mm)*
AR	-	-	-	8/7/1992	22	270
AR+9%	-	-	-	8/7/1992	21	365
AR+18%	-	-	-	8/7/1992	21	405
AR+SA	-	-	-	8/14/1992	108	162
AR+9%+SA	-	-	-	8/14/1992	105	168
AR+18%+SA	-	-	-	8/14/1992	102	166
AR+Age	7/6/1992	27	500	8/28/1992	24	485
AR+9%+Age	-	-	-	8/28/1992	25	480
AR+18%+Age	-	-	-	8/28/1992	29	507
AR+SA+Age	-	-	-	9/16/1992	92	463
AR+9%+SA+Age	-	-	-	9/16/1992	104	457
AR+18%+SA+Age	-	-	-	9/16/1992	128	445

Table 7. Incoloy 908 1/4" plate grain size and microhardness averaged across the three orthogonal planes.

Condition:	1987 heat* memo date	Y9210K nominal grain size *(micrometers)*	HV (kg/sq.mm)	1992 heat memo date	Y9400K nominal grain size *(micrometers)*	HV (kg/sq.mm)
AR	-	-	-	8/7/1992	21±1	268±5
AR+9%	-	-	-	8/7/1992	21±1	352±13
AR+18%	-	-	-	8/7/1992	22±3	375±30
AR+SA	-	-	-	8/14/1992	100±12	162±1
AR+9%+SA	-	-	-	8/14/1992	105±6	170±2
AR+18%+SA	-	-	-	8/14/1992	104±2	165±2
AR+Age	7/6/1992	28±3	499±7	8/28/1992	25±1	484±3
AR+9%+Age	-	-	-	8/28/1992	25±1	484±5
AR+18%+Age	-	-	-	8/28/1992	30±4	506±4
AR+SA+Age	-	-	-	9/16/1992	91±28	434±29
AR+9%+SA+Age	-	-	-	9/16/1992	100±13	458±27
AR+18%+SA+Age	-	-	-	9/16/1992	128±12	430±15

Legend:
AR = As Received
9% = 9% Cold Worked
18% = 18% Cold Worked
SA = Solution Annealed at 1050°C for one hour
Age = Heat Treated at 650°C for 200 hours
* 1987 specimen was flash welded
Vickers microhardness measured with a 300 gram load.

Appendix C: Microstructural data and Photomicrographs

Title:	Incoloy 908 Database Key		
Material history	1/4" Incoloy 908 plate	**Percent cold work:**	Area reduction
Heat number	INCO Heat #	**Anneal:**	Temp & time
Analysis date	Date analysis completed	**Heat treatment**	Temp & time

Photomicrograph

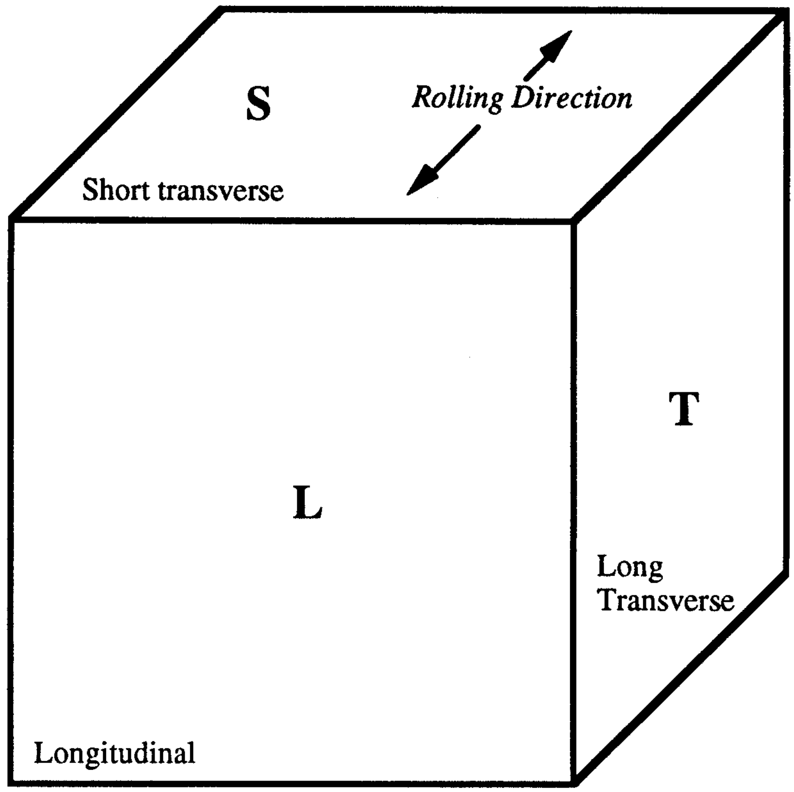


Photo magnification	True magnification	**Etching time in seconds**	
Longitudinal grain size	(microns)	**Longitudinal microhardness**	Vickers scale
Short transverse grain size	(microns)	**Short transverse microhardness**	Vickers scale
Long transverse grain size	(microns)	**Long transverse microhardness**	Vickers scale
Yield Strength at 4K	(MPa)	Ultimate Tensile Strength at 4K	(MPa)
Yield Strength at 77K	(MPa)	Ultimate Tensile Strength at 77K	(MPa)
Yield Strength at 298K	(MPa)	Ultimate Tensile Strength at 298K	(MPa)

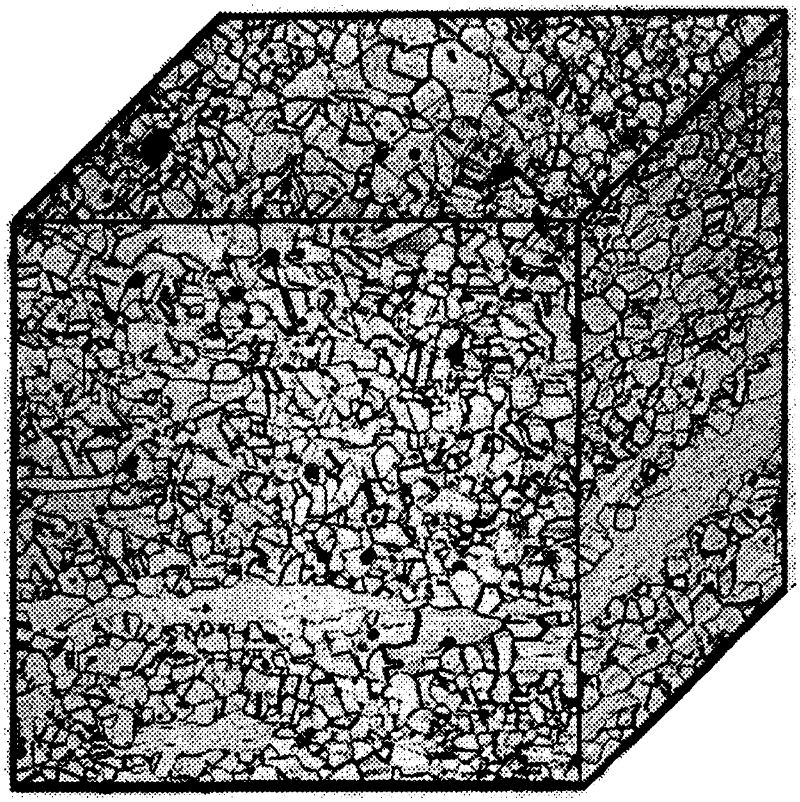
Etchant: All etching of Incoloy 908 for this database used a three-acid stainless steel etchant:

10 ml nitric acid — *10 ml acetic acid /*
15 ml hydrochloric acid — *6 drops glycerol*

Title: As received Incoloy 908 plate

Material history	1/4" Incoloy 908 plate	**Percent cold work:**	0
Heat number	Y9400K (1992)	**Anneal:**	None
Analysis date	08/07/1992	**Heat treatment**	None

Photomicrograph



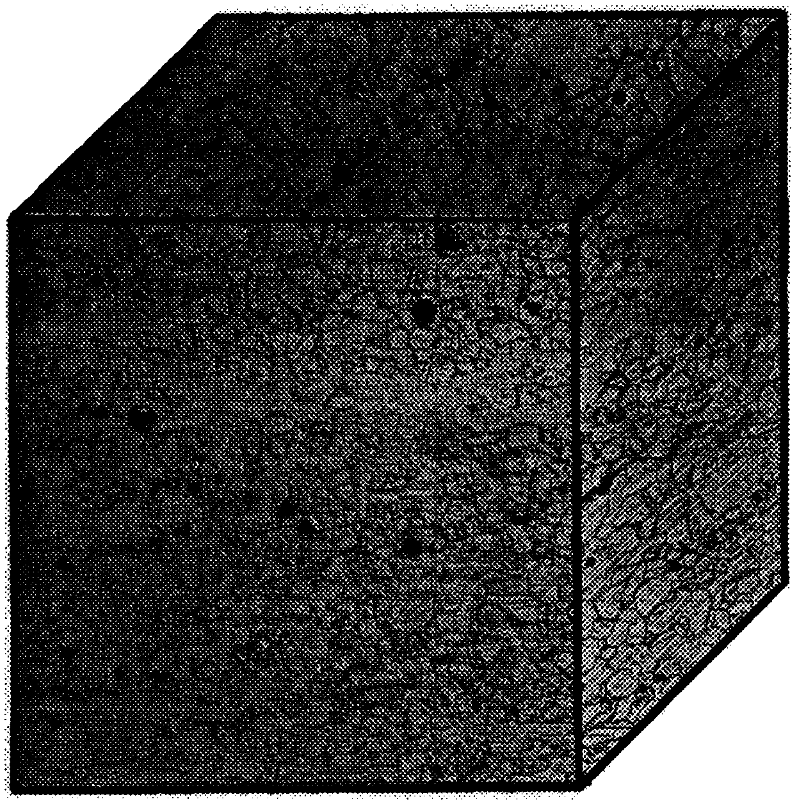
Scale: 100μ

Photo magnification	100x	**Etching time in seconds**	30
Longitudinal grain size	21	**Longitudinal microhardness**	263
Short transverse grain size	20	**Short transverse microhardness**	270
Long transverse grain size	22	**Long transverse microhardness**	270
yield strength 4K in MPa		**UTS 4K in MPa**	
yield strength 77K in MPa		**UTS 77K in MPa**	
yield strength 298K in MPa		**UTS 298K in MPa**	

Title: As received and 9% cold worked Incoloy 908

Material history	1/4" Incoloy 908 plate	**Percent cold work:**	9
Heat number	Y9400K (1992)	**Anneal:**	None
Analysis date	08/07/1992	**Heat treatment**	None

Photomicrograph



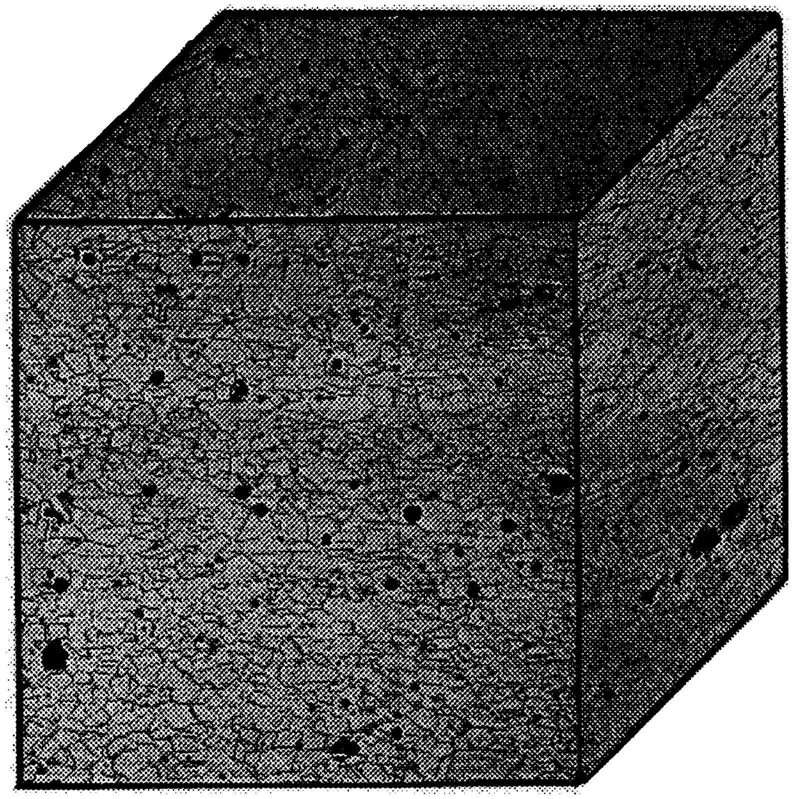
Scale: 100µ

Photo magnification	100x	**Etching time in seconds**	30
Longitudinal grain size	20	**Longitudinal microhardness**	334
Short transverse grain size	21	**Short transverse microhardness**	359
Long transverse grain size	21	**Long transverse microhardness**	365
yield strength 4K in MPa		**UTS 4K in MPa**	
yield strength 77K in MPa		**UTS 77K in MPa**	
yield strength 298K in MPa		**UTS 298K in MPa**	

Title: As received and 18% cold worked Incoloy 908 plate

Material history	1/4" Incoloy 908 plate	**Percent cold work:**	18
Heat number	Y9400K (1992)	**Anneal:**	None
Analysis date	08/07/1992	**Heat treatment**	None

Photomicrograph



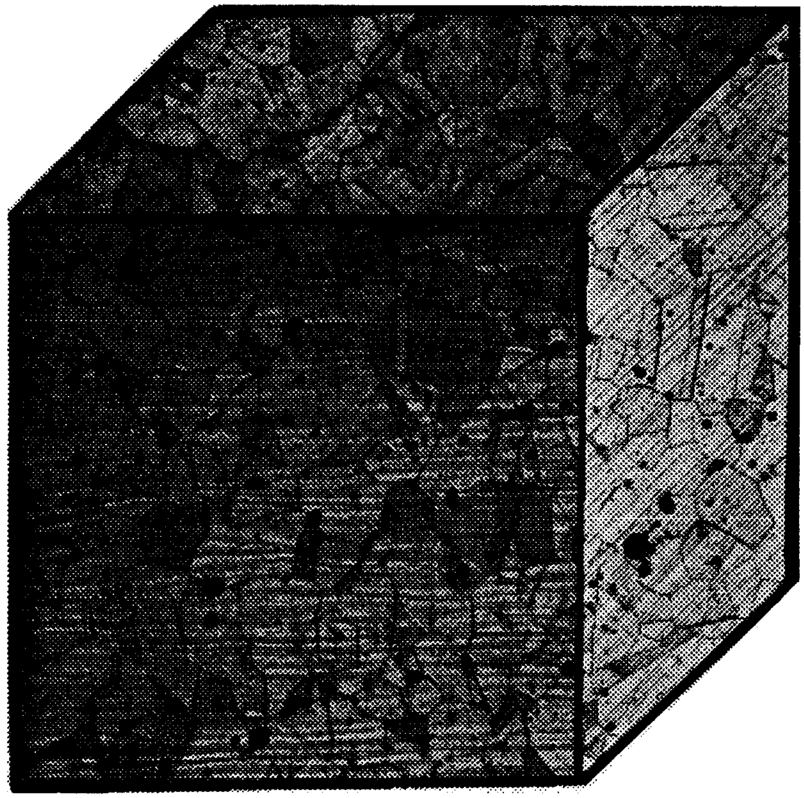
Scale: 100μ

Photo magnification	100x	**Etching time in seconds**	30
Longitudinal grain size	21	**Longitudinal microhardness**	362
Short transverse grain size	25	**Short transverse microhardness**	357
Long transverse grain size	21	**Long transverse microhardness**	405
yield strength 4K in MPa		**UTS 4K in MPa**	
yield strength 77K in MPa		**UTS 77K in MPa**	
yield strength 298K in MPa	947	**UTS 298K in MPa**	1102

Title: As received, 0% cold worked, solution annealed Incoloy 908 plate

Material history	1/4" Incoloy 908 plate	**Percent cold work:**	0
Heat number	Y9400K (1992)	**Anneal:**	1050°C 1 hour
Analysis date	08/14/1992	**Heat treatment**	None

Photomicrograph



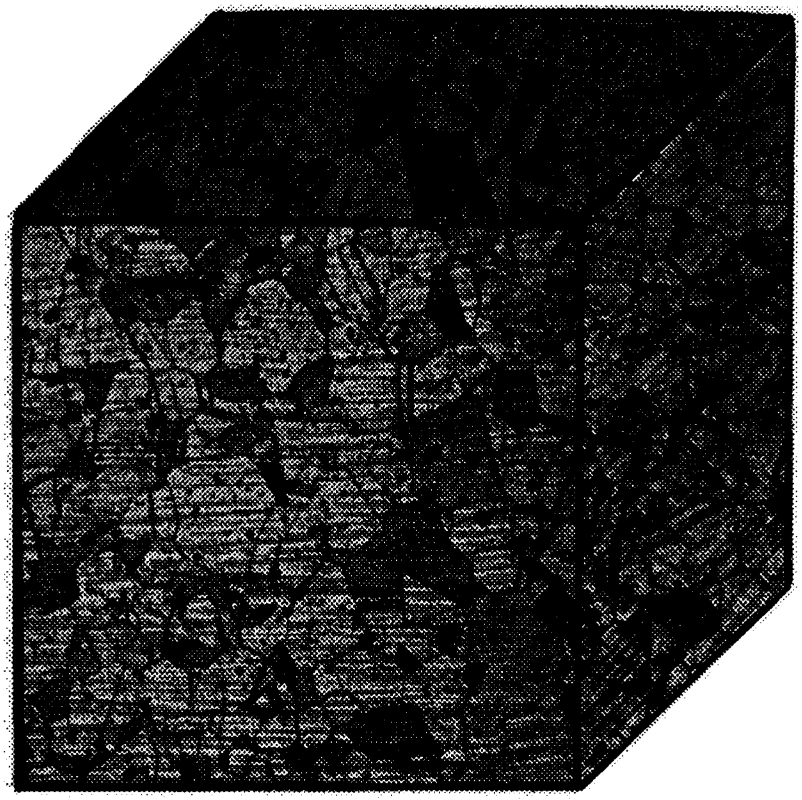
Scale: 200μ

Photo magnification	50x	**Etching time in seconds**	45
Longitudinal grain size	105	**Longitudinal microhardness**	161
Short transverse grain size	88	**Short transverse microhardness**	163
Long transverse grain size	108	**Long transverse microhardness**	162
yield strength 4K in MPa		**UTS 4K in MPa**	
yield strength 77K in MPa		**UTS 77K in MPa**	
yield strength 298K in MPa	389	**UTS 298K in MPa**	717

Title: As received, 9% cold worked, solution annealed Incoloy 908 plate

Material history	1/4" Incoloy 908 plate	**Percent cold work:**	9
Heat number	Y9400K (1992)	**Anneal:**	1050°C 1 hour
Analysis date	08/14/1992	**Heat treatment**	None

Photomicrograph



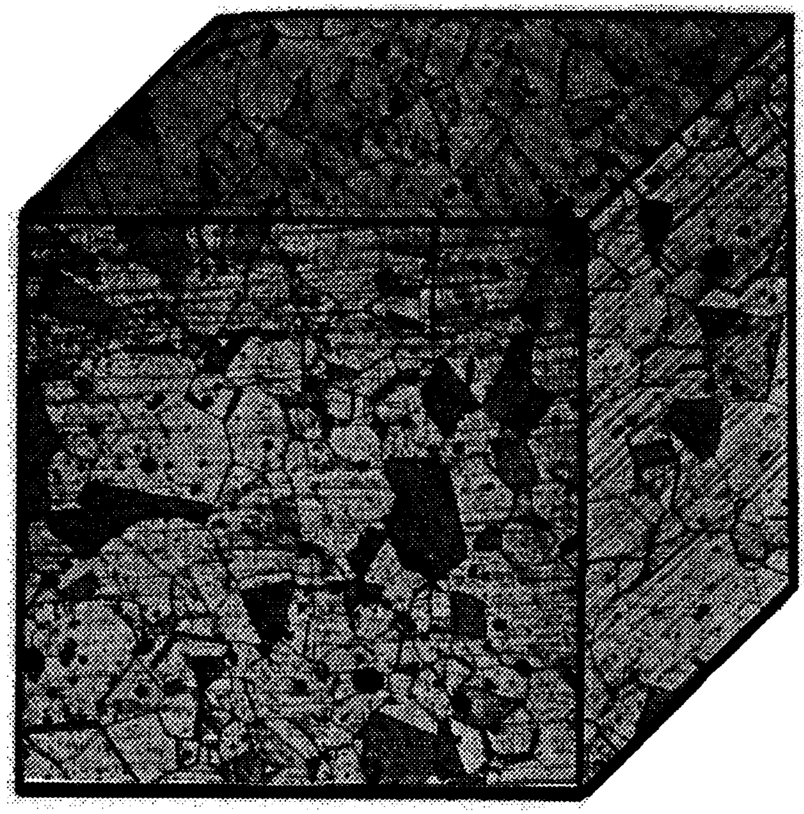
Scale: 200μ

Photo magnification	50x	**Etching time in seconds**	45
Longitudinal grain size	99	**Longitudinal microhardness**	172
Short transverse grain size	110	**Short transverse microhardness**	169
Long transverse grain size	105	**Long transverse microhardness**	168
yield strength 4K in MPa		**UTS 4K in MPa**	
yield strength 77K in MPa		**UTS 77K in MPa**	
yield strength 298K in MPa		**UTS 298K in MPa**	

Title: As received, 18% cold worked and solution annealed Incoloy 908 plate

Material history	1/4" Incoloy 908 plate	**Percent cold work:**	18
Heat number	Y9400K (1992)	**Anneal:**	1050°C 1 hour
Analysis date	08/14/1992	**Heat treatment**	None

Photomicrograph



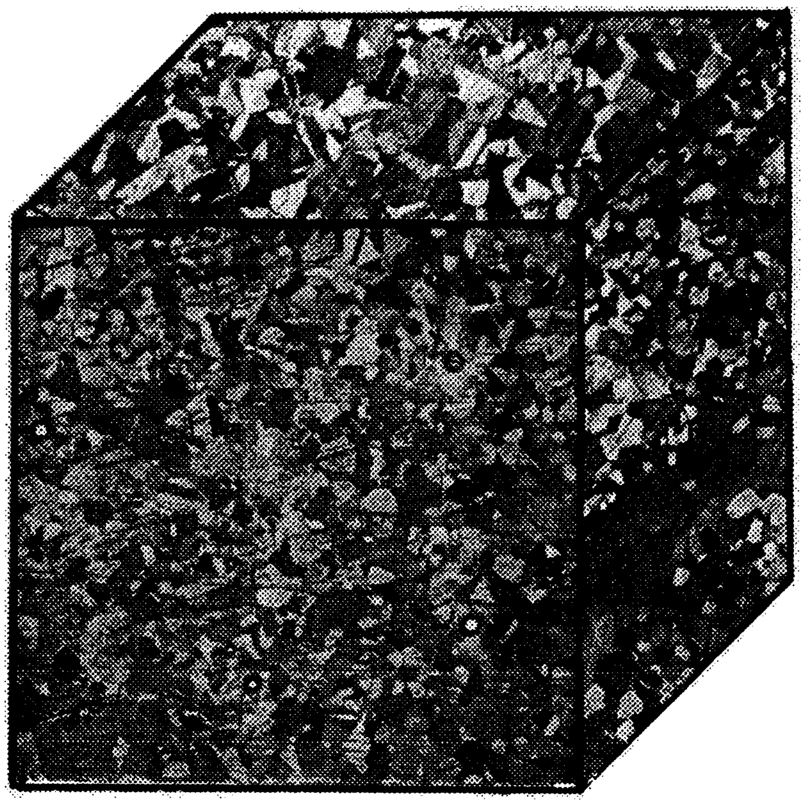
Scale: 200μ

Photo magnification	50x	**Etching time in seconds**	45
Longitudinal grain size	106	**Longitudinal microhardness**	163
Short transverse grain size	105	**Short transverse microhardness**	166
Long transverse grain size	102	**Long transverse microhardness**	166
yield strength 4K in MPa		**UTS 4K in MPa**	
yield strength 77K in MPa		**UTS 77K in MPa**	
yield strength 298K in MPa		**UTS 298K in MPa**	

Title: As received, 0% cold worked and aged Incoloy 908 plate

Material history	1/4" Incoloy 908 plate	**Percent cold work:**	0
Heat number	Y9400K (1992)	**Anneal:**	None
Analysis date	08/28/1992	**Heat treatment**	650°C for 200 hours

Photomicrograph



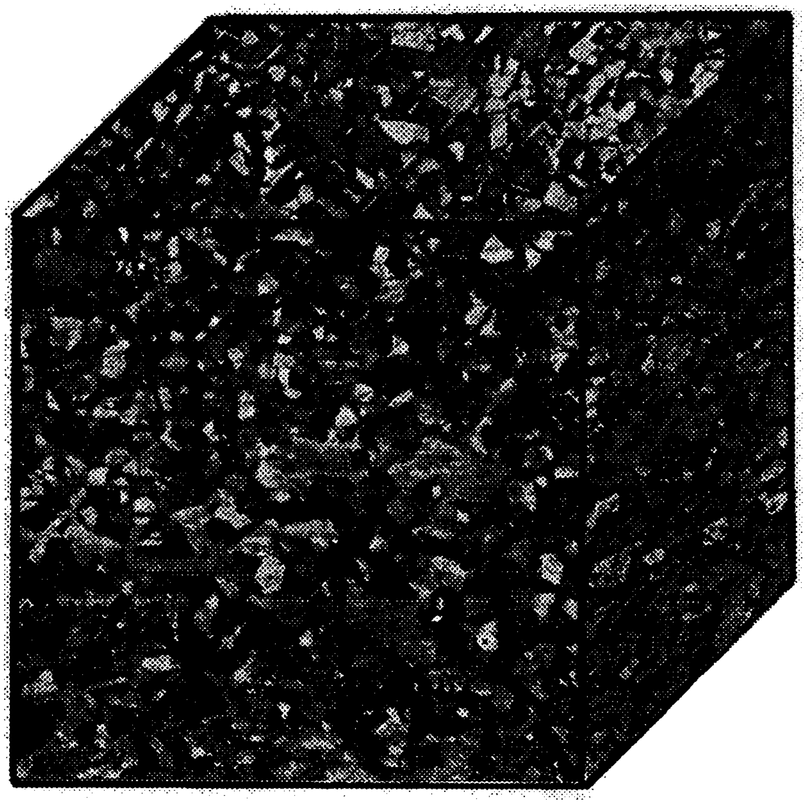
Scale: 100μ

Photo magnification	100x	**Etching time in seconds**	30
Longitudinal grain size	25	**Longitudinal microhardness**	481
Short transverse grain size	24	**Short transverse microhardness**	485
Long transverse grain size	24	**Long transverse microhardness**	485
yield strength 4K in MPa		**UTS 4K in MPa**	
yield strength 77K in MPa		**UTS 77K in MPa**	
yield strength 298K in MPa		**UTS 298K in MPa**	

Title: As received, 9% cold worked and aged Incoloy 908 plate

Material history	1/4" Incoloy 908 plate	**Percent cold work:**	9
Heat number	Y9400K (1992)	**Anneal:**	None
Analysis date	08/28/1992	**Heat treatment**	650°C for 200 hours

Photomicrograph



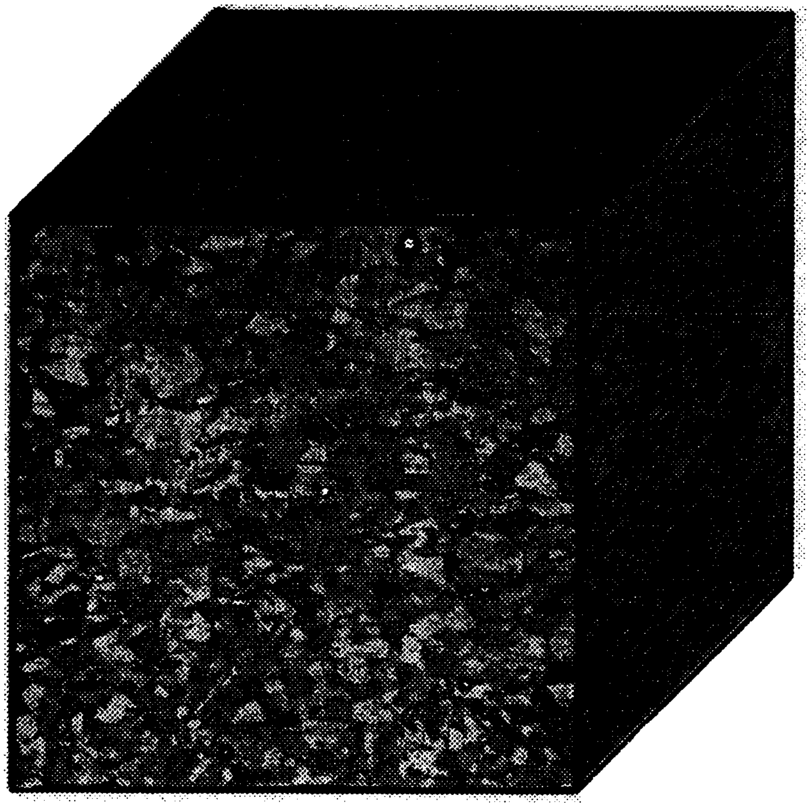
Scale: 100μ

Photo magnification	100x	**Etching time in seconds**	30
Longitudinal grain size	25	**Longitudinal microhardness**	483
Short transverse grain size	27	**Short transverse microhardness**	489
Long transverse grain size	25	**Long transverse microhardness**	480
yield strength 4K in MPa		**UTS 4K in MPa**	
yield strength 77K in MPa		**UTS 77K in MPa**	
yield strength 298K in MPa		**UTS 298K in MPa**	

Title: As received, 18% cold worked and aged Incoloy 908 plate

Material history	1/4" Incoloy 908 plate	**Percent cold work:**	18
Heat number	Y9400K (1992)	**Anneal:**	None
Analysis date	08/28/1992	**Heat treatment**	650°C for 200 hours

Photomicrograph



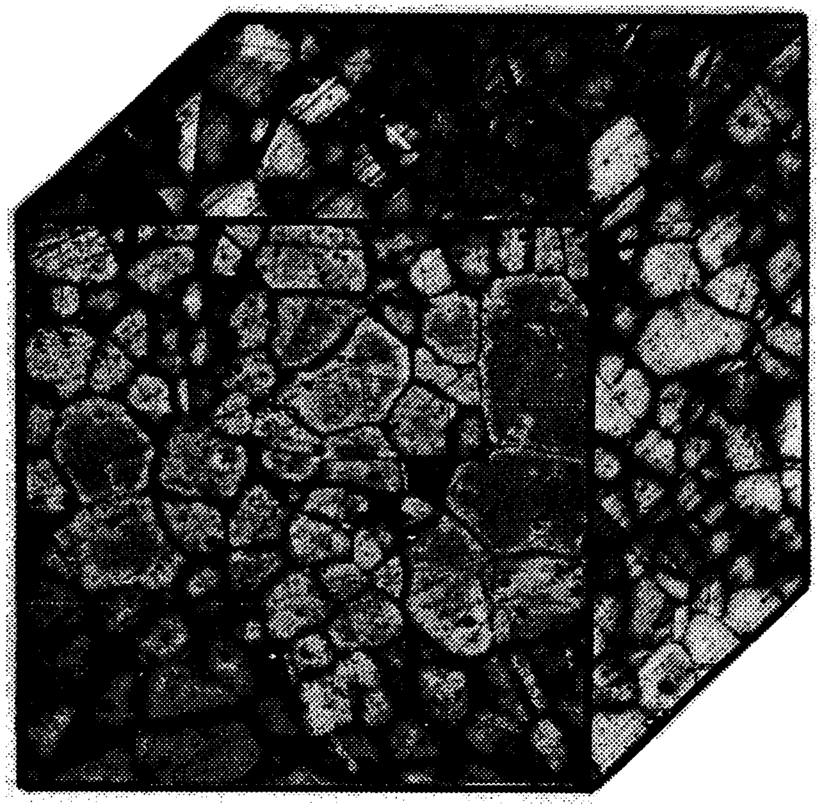
Scale: 100µ

Photo magnification	100x	**Etching time in seconds**	30
Longitudinal grain size	28	**Longitudinal microhardness**	502
Short transverse grain size	34	**Short transverse microhardness**	508
Long transverse grain size	29	**Long transverse microhardness**	507
yield strength 4K in MPa	1489	**UTS 4K in MPa**	1906
yield strength 77K in MPa		**UTS 77K in MPa**	
yield strength 298K in MPa	1279	**UTS 298K in MPa**	1499

Title: As received, 0% cold worked, solution annealed and aged Incoloy 908 plate

Material history	1/4" Incoloy 908 plate	**Percent cold work:**	0
Heat number	Y9400K (1992)	**Anneal:**	1050°C for 1 hour
Analysis date	09/16/1992	**Heat treatment**	650°C for 200 hours

Photomicrograph



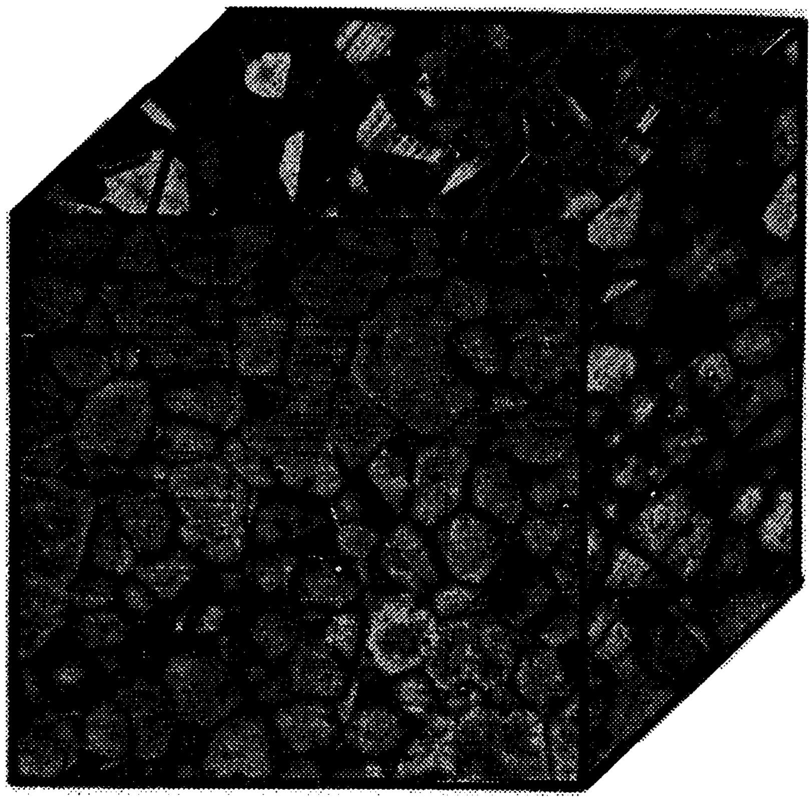
Scale: 200μ

Photo magnification	50x	**Etching time in seconds**	30
Longitudinal grain size	119	**Longitudinal microhardness**	407
Short transverse grain size	79	**Short transverse microhardness**	436
Long transverse grain size	92	**Long transverse microhardness**	463
yield strength 4K in MPa	1070	**UTS 4K in MPa**	1780
yield strength 77K in MPa		**UTS 77K in MPa**	
yield strength 298K in MPa	961	**UTS 298K in MPa**	1354

Title: As received, 9% cold worked, solution annealed and aged Incoloy 908 plate

Material history	1/4" Incoloy 908 plate	**Percent cold work:**	9
Heat number	Y9400K (1992)	**Anneal:**	1050°C for 1 hour
Analysis date	09/16/1992	**Heat treatment**	650°C for 200 hours

Photomicrograph



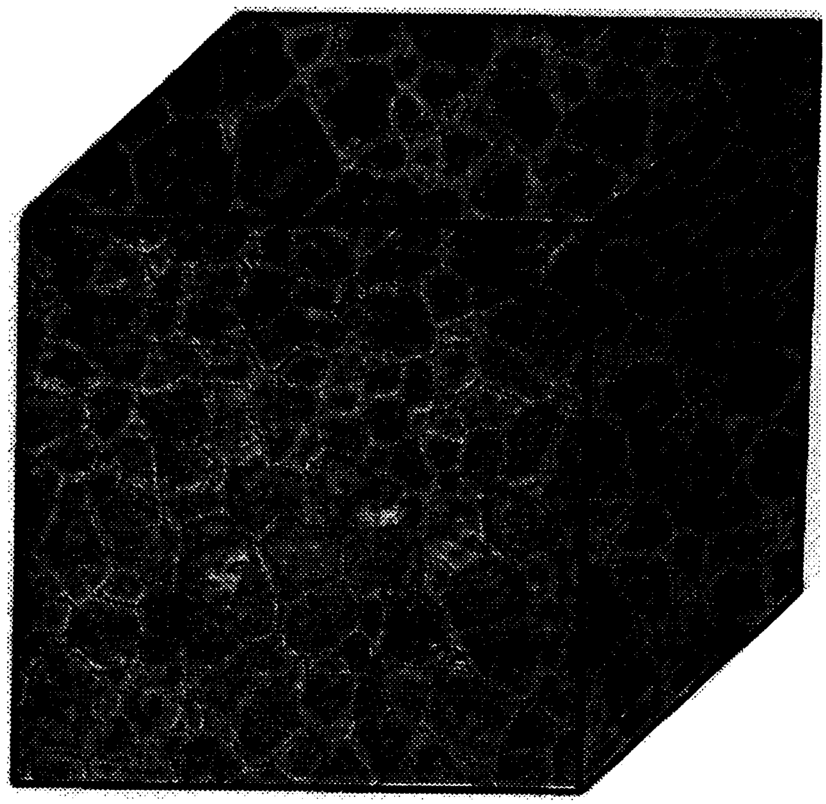
Scale: 200μ

Photo magnification	50x	**Etching time in seconds**	30
Longitudinal grain size	111	**Longitudinal microhardness**	433
Short transverse grain size	87	**Short transverse microhardness**	485
Long transverse grain size	104	**Long transverse microhardness**	457
yield strength 4K in MPa		**UTS 4K in MPa**	
yield strength 77K in MPa		**UTS 77K in MPa**	
yield strength 298K in MPa		**UTS 298K in MPa**	

Title: As received, 18% cold worked, solution annealed and aged Incoloy 908 plate

Material history	1/4" Incoloy 908 plate	**Percent cold work:**	18
Heat number	Y9400K (1992)	**Anneal:**	1050°C for 1 hour
Analysis date	09/16/1992	**Heat treatment**	650°C for 200 hours

Photomicrograph



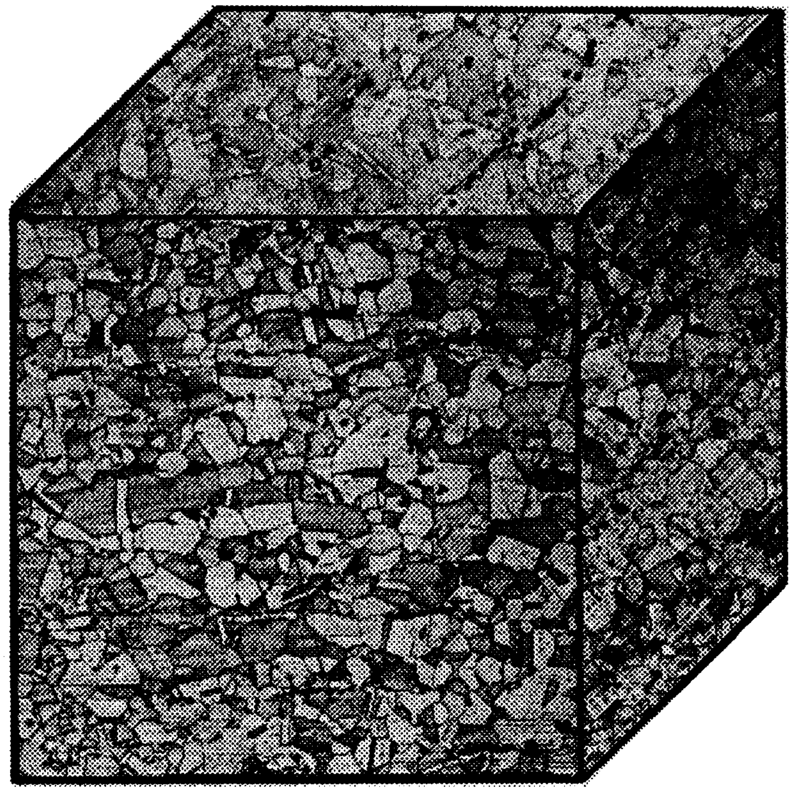
Scale: 200µ

Photo magnification	50x	**Etching time in seconds**	30
Longitudinal grain size	117	**Longitudinal microhardness**	422
Short transverse grain size	134	**Short transverse microhardness**	424
Long transverse grain size	128	**Long transverse microhardness**	445
yield strength 4K in MPa		**UTS 4K in MPa**	
yield strength 77K in MPa	1070	**UTS 77K in MPa**	1680
yield strength 298K in MPa		**UTS 298K in MPa**	

Title: As received, 0% cold worked and aged Incoloy 908 plate

Material history	1/4" Incoloy 908 plate	**Percent cold work:**	0
Heat number	Y9210K (1987)	**Anneal:**	None
Analysis date	07/06/1992	**Heat treatment**	650°C for 200 hours

Photomicrograph



Scale: 100μ

Photo magnification	100x	**Etching time in seconds**	15
Longitudinal grain size	25	**Longitudinal microhardness**	503
Short transverse grain size	31	**Short transverse microhardness**	502
Long transverse grain size	27	**Long transverse microhardness**	500
yield strength 4K in MPa		**UTS 4K in MPa**	
yield strength 77K in MPa		**UTS 77K in MPa**	
yield strength 298K in MPa		**UTS 298K in MPa**	

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