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# Modification of Ni-Cr Base Alloys for Steam Generator Tubes and Other Parts in Pressurized Boiling Water Reactors

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***Abstract*: The degradation and deterioration of structural parts of Reactors in operation in Pressurized and Boiling water Reactors calls for development of special alloy steels, Nickel-Chromium base alloys.**

**The Ni – Cr alloys are well-known for their capability to maintain metallographic structure at high temperatures and for withstanding thermal stresses caused due to continuous exposure of heat in usage. The Incoloy -800 alloys are found meeting requirements in this regard.**

**Like stainless steels these are chosen for components exposed to primary reactor for their strength and corrosion resistance at temperature up to 345Oc. These NI-Cr-Fe alloys withstand high temperature oxidation. However, due to the cost of nickel base alloys relative to stainless steels, the former are only chosen where specific circumstances dictate that no stainless steels alternative is suitable.**

**The chemical composition of Ni-Cr alloy is as follows.**

**Ni = 30-36% Cr = !9-23% S= 0.015%**
**Al = 0.15-0.6% Ti = 0.15-0.6% Cu= 0.75%**
**C=0.1% max Si=1% Mn= 1.5%**
**Fe = balance**

***The chromium in alloy gives resistance to oxidation and corrosion.**

*** The Nickel content provides resistance to scaling, general corrosion and resistance due to stress corrosion cracking. The high percentage Nickel content also helps in retaining austenitic structure so that the alloy is ductile. The iron Fe content provides resistance to internal oxidation.**

**In this work we study development and manufacturing of alloy steel Incoloy-800, required for manufacture of SG tubes, which are being processed at Nuclear Fuel Complex.**

**Incoloy800 is widely used alloy steel in areas requiring corrosion resistance, heat resistance, strength and stability for service upto 816$^{O}$C. This alloy steel offers general corrosion resistance to many aqueous media and by virtue of its nickel content also resists stress corrosion cracking.**

**At elevated temperatures, it offers resistance to oxidation, carburization and sulphidation along with rupture and creep strength. Because of these reasons, this alloy is an important material in production of steam generator tubing in Nuclear power plants.**

**At Nuclear Fuel Complex, INCOLOY-800 alloy is produced in Arc Furnaces and Billets are manufactured.**

**The alloy elements are initially mixed in proper composition and then compacted at high pressure. The compacted alloy elements along with Fe are heated and melted in Electric arc furnace. After obtaining required composition in the melt, the molten metal is poured in form of Billets.**

**The samples are taken from the billet, from the tube extruded and from tube after final pillgaring.**

**These Billets are extruded to form tube form. Later in several stages of operation of extrusion the tubes of required thickness and length are manufactured.**

**The obtained samples are then analyzed for metallurgical structure and for mechanical properties as required for Incoloy-800 alloy.**

***Keywords*: Incoloy-800, Nickel-Chromium base alloys, NI-Cr-Fe alloys, Nuclear Fuel Complex, pillgaring, stress corrosion cracking.**

## 1. Introduction

The degradation and deterioration of structural parts of Reactors in operation in Pressurised and Boiling water Reactors calls for development of special alloy steels, Nickel-Chromium base alloys.

The Ni – Cr alloys are well-known for their capability to maintain metallographic structure at high temperatures and for withstanding thermal stresses caused due to continuous exposure of heat in usage. The Incoloy -800 alloys are found meeting requirements in this regard.

Like stainless steels these are chosen for components exposed to primary reactor for their strength and corrosion resistance at temperature up to 345$^{O}$c. These NI-Cr-Fe alloys withstand high temperature oxidation. However, due to the cost of nickel base alloys relative to stainless steels, the former are only chosen where specific circumstances dictate that no

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stainless steels alternative is suitable.

Three broad categories of use of Nickel base alloy are as follows

1. As interface materials between Carbon and Low Alloy Steels (C&LAS) and SSs;
2. As corrosion resistant steam generator tubing in PWR;
3. As high strength fasteners and springs immersed in LWR coolants.

Table1: Thermal conductivity and thermal coefficient of Alloys 600 and 690 compared to Alloy 800 austenitic SS and low ally steel are shown below.

## 2. Mechanical Properties

The major differences between alloys 800, 800H and 800HTare mechanical properties. The differences stem from the restricted compositions of alloys 800H and 800HT and the high-temperature anneals used for these alloys. In general, alloy 800 has higher mechanical properties at room temperature and during short-time exposure to elevated temperatures, whereas alloys 800H and 800HT have superior creep and rupture strength during extended high temperature exposure.

### *A. Tensile Properties*

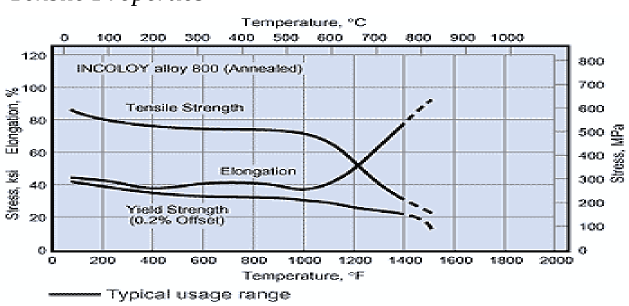



Fig. 1. High-temperature strength tensile properties of Incoloy-800

| Temperature | Tensile Modulus | Shear Modulus | Poisson's Ratio[b] |
|---|---|---|---|
| °F | $10^3$ ksi | $10^3$ ksi | |
| -310 | 30.55 | 11.45 | 0.334 |
| 75 | 28.50 | 10.64 | 0.339 |
| 200 | 27.82 | 10.37 | 0.341 |
| 400 | 26.81 | 9.91 | 0.353 |
| 600 | 25.71 | 9.47 | 0.357 |
| 800 | 24.64 | 9.04 | 0.363 |
| 1000 | 23.52 | 8.60 | 0.367 |
| 1200 | 22.37 | 8.12 | 0.377 |
| 1400 | 21.06 | 7.58 | 0.389 |
| 1600 | 19.20 | 6.82 | 0.408 |
| °C | GPa | GPa | Poisson's Ratio[b] |
| -190 | 210.6 | 78.9 | 0.334 |
| 20 | 196.5 | 73.4 | 0.339 |
| 100 | 191.3 | 71.2 | 0.343 |
| 200 | 184.8 | 68.5 | 0.349 |
| 300 | 178.3 | 66.1 | 0.357 |
| 400 | 171.6 | 63.0 | 0.362 |
| 500 | 165.0 | 60.3 | 0.367 |
| 600 | 157.7 | 57.4 | 0.373 |
| 700 | 150.1 | 54.3 | 0.381 |
| 800 | 141.3 | 50.7 | 0.394 |

### *B. Tensile Properties*

Low-cycle fatigue strength of alloys 800, 800H and 800HTat room temperature and 1400°F (760°C). Low-cycle fatigue data for alloys 800, 800H and 800HTare compared at 1000°F (538°C) and 1200°F (649°C).

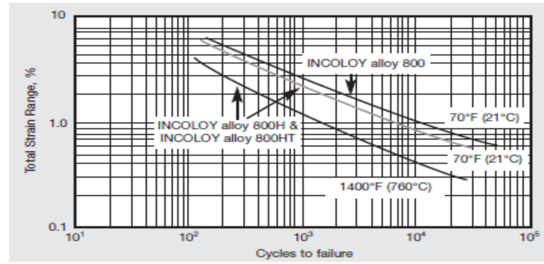



Fig. 2. Low-cycle fatigue strength of Incoloy-800

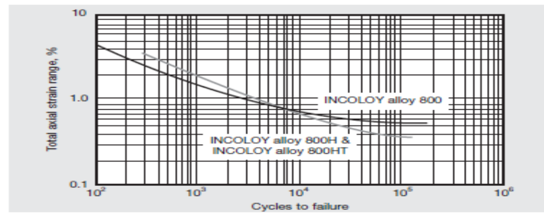



Fig. 3. Low-cycle fatigue strength of Incoloy 800 at 1000°F (540°C)

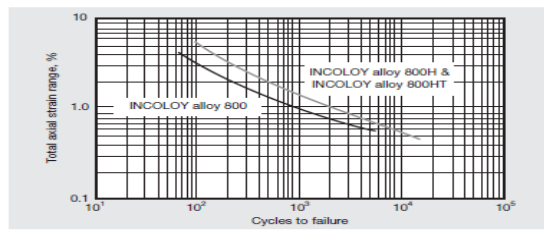



Fig. 4. Low-cycle fatigue strength of Incoloy 800 at 1200°F (650°C).

### *C. Creep and Rupture Properties*

The outstanding characteristics of both Incoloy alloys 800 are their high creep and rupture strengths. The controlled chemistries and solution annealing treatment are designed to produce optimum creep-rupture properties. Figure shows creep strength of alloys 800H and 800HT at various temperatures.

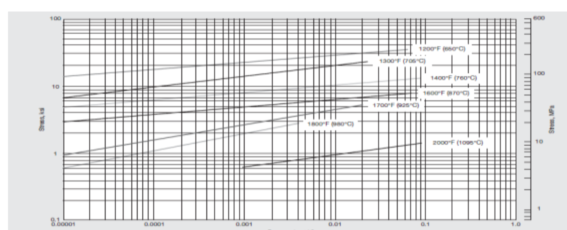


Fig. 5. Typical creep strength of INCOLOY alloys 800

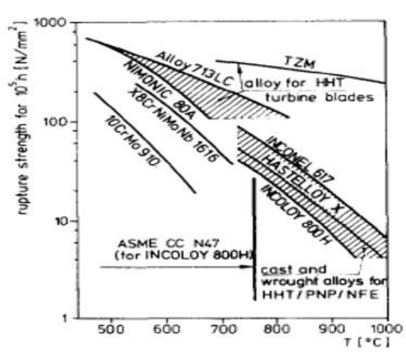



Fig. 6. Creep ruptures strength of different materials

### *D. Corrosion Resistance*

Alloys 800, 800H and 800HT have the same nickel, chromium, and iron contents and generally display similar corrosion resistance. Since alloys 800 are used for their high-temperature strength, corrosive environments to which these alloys are exposed normally involve high temperature reactions such as oxidation and carburization.

Table 1
Corrosion rates in refinery furnace atmosphere

| Alloy | Corrosion Rate (mm/y) |
|---|---|
| INCOLOY alloys 800 | 0.15 |
| Type 310 Stainless Steel | 0.23 |
| Type 309 Stainless Steel | 2.15 |
| Type 304 Stainless Steel | Complete oxidation |

## 3. Heat treatment

### *A. Annealing - Basic Practice*

Specific annealing procedures for Incoloy alloys 800 depend on the amount of cold work, intended grain size and cross section of the material. The mechanical properties of heavily cold worked material are only slightly affected by temperatures below 1000°F (540°C). Stress relief begins at about 1000°F (540°C) and is virtually complete after 1600°F (870°C) for a time commensurate with thickness. As an example, a general guideline for stress relief for plate products would be 1 hour per inch (25mm) of thickness or 1½ hours at 1600°F (870°C), whichever is the greater. A stress relief will generally require more time than recrystallization anneal. The effect of cold work in reducing their crystallization temperature for Incoloy alloys 800Hand 800HT strip. Time at temperature was 30 minutes. The lower curve indicates the temperature when recrystallization began, and the upper curve when complete. Intermediate

Temperatures will usually result in a fine recrystallized structure interspersed with a cold-worked, elongated grain structure. Temperatures above the upper curve will cause grain growth.

These alloys are designed for high-temperature service. Optimum resistance to time-dependent deformation (creep)at elevated temperatures is obtained by heating to at temperature to cause grain growth. The temperature normally used is 2100 to 2200°F (1150–1200°C). Depending on the size and furnace characteristics, the time at temperature is adjusted to achieve a grain size of ASTM No. 5 or coarser. The temperature and time should also be adjusted to limit excessive grain growth since little additional creep strength is obtained as additional grain growth occurs. One disadvantage of excessive grain grows this the lowering of toughness after exposure to elevated temperature. Material that will be cold formed more than about 20%should be ordered in the fine-grain condition. Material that will be heated for hot working should be in the as hot finished condition or as-annealed. For optimum strength after fabrication, the material should be annealed as indicated above to obtain a minimum average grain size of ASTM No. 5. One advantage in deforming fine grain material is the reduced surface roughness commonly called "orange peel". Another is the reduced thermal cracking tendency of the fine grain versus the coarse grain material. Highly cold-worked components having shapes that do not allow spring back are especially susceptible to cracking when heated. The driving force for these tight cracks is a high residual tensile stress. The fine grain material will relax residual stresses more rapidly when heated to the annealing temperature, thus reducing the tendency for cracking. At times, it is not possible to heat treat after fabrication because of component size or economics. The following are guidelines for applications where coarse grain material is placed in elevated temperature service. One is to limit cold work to less than 20% strain and another is to limit the service temperature and duration so as not to cause recrystallization. The beginning of recrystallization after cold straining 10 and 20% versus annealing time or service duration. This figure presents only an approximation of the temperature and duration limit since the compositional variations from heat to heat and the thermo mechanical history involved will influence recrystallization behavior. In summary, post fabrication heat treatments depend on the amount of resulting strain from the fabrication (forming and/or welding) and the service conditions. From this, and the data contained in Figures 10, 11, 12, and 13 one can determine whether to use the 1600°F (870°C) stress relieving temperature or the 2100°F (1150°C) minimum solution annealing temperature when conducting post fabrication heat treatments.

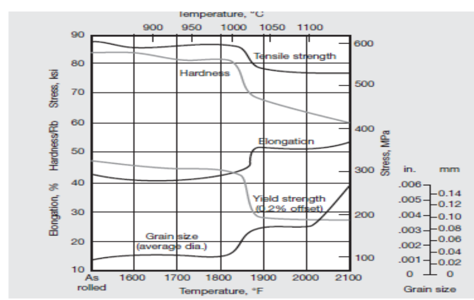



Fig. 7. Effect of annealing temperatures on properties of Incoloy-800

### *B. Oxidation*

Because of their high chromium and nickel contents, Incoloy alloys 800 have excellent resistance to oxidation. The chromium in these alloys promotes the formation of a protective surface oxide, and the nickel enhances the protection, especially during cyclic exposure to high temperatures.

The tests were conducted in air and consisted of alternating exposure to temperatures for 15 minutes and cooling in still air for 5 minutes. The specimens were subjected to 1000 h of cyclic exposure with periodic removal for weight change measurements. The furnace operated at 600°F to 2100°F (870-1150°C) and was fired by fuel having no sulfur. The samples were exposed in the furnace for 3 months. In atmospheres that are oxidizing to chromium but reducing to nickel, nickel-chromium alloys may be subject to internal oxidation. The condition, which causes severe embrittlement, is characterized by extensive oxidation of chromium, leaving the remaining metal strongly magnetic. Susceptibility to internal oxidation is decreased by the addition of iron to nickel-chromium alloys. Incoloy alloys 800, with 46% iron, are resistant to internal oxidation.

*C. Carburization*

The high nickel content of alloys 800 provides good resistance to carburizing environments. Table the resistance to carburizing atmospheres at 1700°F (925°C) and 1800°F (980°C) for these alloys. The test atmosphere consisted of 2% methane hydrogen. Compares Incoloy alloys 800H and 800HTwith some other alloys having high resistance to carburization. The atmosphere was composed of 2%methane and 5% argon in hydrogen.

*D. Sulfidation*

Because of their high chromium content, alloys 800H have good resistance to many sulfur-containing atmospheres at high temperatures. Table of sulfidation tests performed at 1110°F (600°C) and 1290°F(700°C) in an atmosphere of 1.5% hydrogen sulfide in hydrogen. The weight-loss measurements are for descaled specimens after 100 h of exposure.

*E. Nitriding*

Studies involving various nitriding environments have shown that the resistance of nickel-iron-chromium alloys to nitriding increases with increasing nickel content. Although INCONEL alloy 600 (76% nickel) is usually preferred fornitriding service, Incoloy alloys 800H and 800HT (32%nickel) have good resistance to many nitriding atmospheres. Table compares alloys 800H and 800HT with several other materials in tests performed in an ammonia converter. The samples were exposed for 3 years to the atmosphere of65% hydrogen and 35% nitrogen at 11 ksi (75.8 MPa) and1000°F (540°C).

*F. Heating and Pickling*

All material to be heated must be clean. Oil, paint, grease, shop soil and other foreign substances must be removed prior to the heating operation. Heating must be performed in a low-sulfur atmosphere. Open heating must be done with low-sulfur fuel, and the furnace atmosphere must be maintained in a reducing condition to prevent excessive oxidation. Because of the readiness with which chromium is oxidized into a refractory oxide by air, carbon dioxide or water vapor, 800-series alloys cannot be bright annealed in the usual industrial annealing furnace. Under closely controlled conditions, the alloy can be bright annealed in dry, pure hydrogen (dew point of -73°F (-58°C) or lower, less than 0.004% by volume water, and less than 0.007% by volume air).

Incoloy alloys 800 are normally annealed in box or muffle furnaces using prepared reducing atmospheres. A satisfactory atmosphere is formed by the products of combustion from low-sulfur natural gas burned with a deficiency of air. It produces a thin, adherent, green black film of oxide on the material. Oxidizing atmospheres produce a heavy black scale that is difficult to remove. Removal of such scale often requires considerable grinding. The alloys usually must be pickled after being heated ifa bright surface is required. Because of the alloy's inherent resistance to chemical attack, specialized pickling procedures are needed.

*G. Hot Working Characteristics*

Proper temperature control during deformation is the most important factor in achieving hot malleability. Preheating all tools and dies to 500°F (260°C) is recommended to avoid chilling the metal during working. Heavy forging should not be done so rapidly that the metal becomes overheated. In hot bending operations, the metal should be worked as soon as possible after removal from the furnace to minimize surface cooling before bending is completed. The hot forming range for alloys 800 is1600–2200°F (870–1200°C). Heavy forging should be done at temperatures down to 1850°F (1010°C) and light working can be accomplished down to 1600°F (870°C). No working should be done between 1200 to 1600°F (650–870°C). The rates of cooling following hot forming is not usually critical for these alloys with respect to thermal cracking. However, they are subject to some carbide precipitation in the 1000 to 1400°F (540–760°C) temperature range and should be rapidly cooled through that range when sensitization is a concern. Cooling after hot working should be air cool or faster. Heavy sections may become sensitized during cooling from the hot-working temperature, and therefore be subject to intergranular corrosion in certain media. Compares the stress-rupture strengths of Electrode 117 and Incoloy alloys 800.

*H. Inconel 600*

Inconel is a registered trademark of special metals Corporation that refers to a family of austenitic nickel-chromium-based super alloys. Inconel alloys are typically used in high temperature applications. It is often referred to in English as "Inco" (or occasionally "Iconel"). Common trade names for Inconel include: Inconel 625, Chronin 625, Altemp 625, Haynes 625, Nickelvac 625 and Nicrofer 6020.

The Inconel family of alloys was first developed in the 1940s by research teams at the Wiggin Works in Hereford, England, in support of the development of the Whittle jet engine.

Chemical Composition
Nickel- 72 % min
Chromium – 14 – 17%
Iron – 6- 10%
Manganese – 1%
Copper – 0.5%
Silicon – 0.5%
Carbon – 0.15%
Sulfur – 0.015%

### I. Properties

Inconel alloys are oxidation and corrosion resistant materials well suited for service in extreme environments. When heated, Inconel forms a thick, stable, passivating oxide layer protecting the surface from further attack. Inconel retains strength over a wide temperature range, attractive for high temperature applications where aluminum and steel would succumb to creep as a result of thermally-induced crystal vacancies (see Arrhenius equation). Inconels high temperature strength is developed by solid solution strengthening or precipitation strengthening, depending on the alloy. In age hardening or precipitation strengthening varieties, small amounts of niobium combine with nickel to form the intermetallic compound $Ni_3Nb$ or gamma prime (γ'). Gamma prime forms small cubic crystals that inhibit slip and creep effectively at elevated temperatures.

Table 2
Properties of Inconel-600

| Property | Metric | Imperial |
|---|---|---|
| Density | 8.47 g/cm³ | o.306 lb/in³ |
| Melting point | 1413°C | 2575°C |
| Co-Efficient of expansion | 13.3 µm/m.°C | $7.4\times10^{-6}$ in/in.°F |
| Modulus of rigidity | 75.6 kN/mm² | 10965 ksi |
| Modulus of elasticity | 206 kN/mm² | 29878 ksi |

### J. Machining

Inconel is a difficult metal to shape and machine using traditional techniques due to rapid work hardening. After the first machining pass, work hardening tends to plastically deform either the work piece or the tool on subsequent passes. For this reason, age-hardened Inconels such as 718 are machined using an aggressive but slow cut with a hard tool, minimizing the number of passes required. Alternatively, the majority of the machining can be performed with the work piece in a solutionised form, with only the final steps being performed after age-hardening. External threads are machined using a lathe to "single point" the threads, or by rolling the threads using a screw machine. Holes with internal threads are made by welding or brazing threaded inserts made of stainless steel. Cutting of plate is often done with a water jet cutter. Internal threads can also be cut by single point method on lathe, or by thread milling on a machining centre. New whisker reinforced ceramic cutters are also used to machine nickel alloys. They remove material at a rate typically 8 times faster than carbide cutters. 718 Inconel can also be roll threaded after full aging by using induction heat to 1300 degrees F without increasing grain size.

### K. Welding

The commonly used welding methods work well with this alloy. Matching alloy filler metal should be used. If matching alloy is not available then the nearest alloy richer in the essential chemistry (Ni, Co, Cr, Mo) should be used. All weld beads should be slightly convex. It is not necessary to use preheating. Surfaces to be welded must be clean and free from oil, paint or crayon marking. The cleaned area should extend at least 2" beyond either side of a welded joint. Gas-Tungsten Arc Welding: DC straight polarity (electrode negative) is recommended. Keep as short an arc length as possible and use care to keep the hot end of filler metal always within the protective atmosphere. Shielded Metal-Arc Welding: Electrodes should be kept in dry storage and if moisture has been picked up the electrodes should be baked at 600 F for one hour to insure dryness. Current settings vary from 60 amps for thin material (0.062" thick) up to 140 amps for material of 1/2" and thicker. It is best to weave the electrode slightly as this alloy weld metal does not tend to spread. Cleaning of slag is done with a wire brush (hand or powered). Complete removal of all slag is very important before successive weld passes and also after final welding. Gas Metal-Arc Welding: Reverse-polarity DC should be used and best results are obtained with the welding gun at 90 degrees to the joint. For Short-Circuiting-Transfer GMAW a typical voltage is 20-23 with a current of 110-130 amps and a wire feed of 250-275 inches per minute. For Spray-Transfer GMAW voltage of 26 to 33 and current in the range of 175-300 amps with wire feed rate of 200-350 inches per minute are typical. Submerged-Arc Welding: Matching filler metal, the same as for GMAW, should be used. DC current with either reverse or straight polarity may be used. Convex weld beads are preferred.

### L. Joining

Welding Inconel alloys is difficult due to cracking and microstructural segregation of alloying elements in the HAZ (heat affected zone). However, several alloys have been designed to overcome these problems. The most common welding method is TIG (Tungsten Inert gas welding). New innovations in pulsed micro laser welding have also become more popular in recent years.

### M. Uses

Inconel is often encountered in extreme environments. It is common in gas turbine blades, seals, and combustors, as well as turbocharger rotors and seals, electric submersible well pump motor shafts, high temperature fasteners, chemical processing and pressure vessels, heat exchanger tubing, steam generators in nuclear pressurized water reactors, natural gas processing with contaminants such as $H_2S$ and $CO_2$,firearm sound suppressor blast baffles, and Formula One and NASCAR exhaust systems. Inconel is increasingly used in the boilers of waste incinerators. The Joint European Torus vessel is made in Inconel. Inside JET plasma is heated to temperatures that are higher than those found in the Sun. A strong magnetic field keeps the plasma away from the vessel. North constructed the skin of the X-15 rocket plane out of an Inconel alloy known as "Inconel X". Rolled Inconel was frequently used as the recording medium by engraving in Black Box recorders on aircraft. Alternatives to the use of Inconel in chemical applications like scrubber, columns, reactors, and pipes is Hastelloy, perfluoroalkoxy (PFA) lined carbon steel or fiber reinforced plastic.

## 4. Production Process of SG Tubing

General specifications for tubes Specifications for tubing size 63mm OD x 9mm WT.

150mm Diameter round billet preparation

↓

142mm outer dia x 50mm inner dia billet

↓

Hot extrusion at 1160°C-1190°C

↓

63mm OD x 9mm WT

↓

Straightening & deglassing

↓

Blank condition
(OD grinding &ID honing)

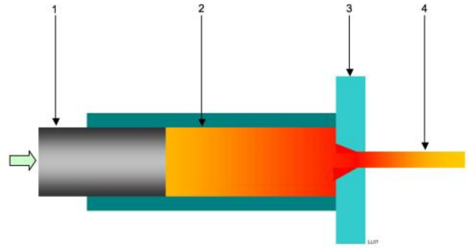
↓

Multiple cold reductions by pillgaring
Final size OD 19.1 x 1.1 WT

### A. Extrusion

For the extrusion of these billets were machined for surface smoothening. The billets are machined to the standard size of having 150mm diameter. When the billet is prepared the surface will doesn't have proper smoothening so it undergoes machining to make the surface smooth, then diameter of the billet is reduced to 142 mm. The billet is heated in the muffle furnace to the temperature 1160-1190°C. The billet is extruded in the extrusion machine.

The billet is jacketed by the glass lubricant, which makes the billet extruded easily. The extrusion is carried out by using horizontal hydraulic direct extrusion press of capacity 3780 tons. Initially the die and the container are pre heated to a temperature of about 200°C, so that the billet doesn't loss much temperature during extrusion. After the billet is obtains the required temperature, the billet is placed in the container along with dummy block and pierced with mandrel into the drill, so that the tube will be extruded without changing the shape. The glass pad is placed at the face of the die in front of the nose of the billet. During extrusion the hot billet progressively softens the glass pad and provides a lubricant film between the extrusion and the die.




1. Ram, 2. Hot billet, 3. Die, 4. Extruded tube
Fig. 8. Extrusion Process

### B. Chemical Analysis

Analytical chemistry is the study of the separation, identification, and quantification of the chemical components of natural and artificial materials. Qualitative analysis gives an indication of the identity of the chemical species in the sample and quantitative analysis determines the amount of one or more of these components. The separation of components is often performed prior to analysis.

Analytical methods can be separated into classical and instrumental. Classical methods (also known as wet chemistry methods) use separations such as precipitation, extraction and distillation and qualitative analysis by color, odor, or melting point. Quantitative analysis is achieved by measurement of weight or volume. Instrumental methods use an apparatus to measure physical quantities of the analyze such a slight absorption, fluorescence, or conductivity. The separation of materials is accomplished using chromatography or electrophoresis methods

### C. Hardness Test

The hardness value of the Incoloy-800 is determined by Vickers hardness method. The Vickers hardness test method consists of indenting the test material with a diamond indenter, in the form of a right pyramid with a square base and an angle of 136 degrees between opposite faces subjected to a load of 1 to 100 kgf. The full load is normally applied for 10 to 15 seconds. The two diagonals of the indentation left in the surface of the material after removal of the load are measured using a microscope and their average calculated. The area of the sloping surface of the indentation is calculated. The Vickers hardness is the quotient obtained by dividing the kgf load by the square mm area of indentation.

$$HV = \frac{2F\sin\frac{136^\circ}{2}}{d^2} \qquad HV = 1.854\frac{F}{d^2}\ \text{approximately}$$

### D. Metallography

Metallography is the science and art of preparing a metal surface for analysis by grinding, polishing and etching to reveal micro structural constituents. After preparation, the sample can easily be analyzed using Olympus optical microscope.

The various steps required to prepare the specimen are as follows, *1) Sample collection*

The samples are collected at various stages during extrusion, sample is collected after the billet is prepared. Then other sample after extrusion of the billet, and other sample from the tube final product after pillgaring.

*2) Rough Grinding*

The specimens were rough ground on a belt sander (grit 80), with the specimens kept cool by frequent dropping in water during the grinding operation. The samples were moved perpendicular to the existing scratches to facilitate the recognition of the stage when the deeper scratches have been replaced by shallower one's characteristic of the finer abrasive. The rough grinding is continued till the surface is flat and free of nicks, burrs etc. and all the scratches due to the hacksaw are no longer visible.

*3) Intermediate Polishing*

The specimens were polished in a disc polisher (grit 600), lubricating with which prevented overheating of the samples, minimized smearing and provided a rinsing action to flush away the surface removal products.

*4) Fine Polishing*

The final approximation to a flat scratch-free surface was obtained by use of a wet rotating wheel covered with velvet as the polishing cloth. Diamond paste was also used as final polishing abrasive. Thus a properly polished scratch-free specimen was obtained.

*5) Etching*

The purpose of etching is to make visible the many structural characteristics of the metal of alloy. The process must be such that the various parts of the microstructure will be clearly differentiated. This is accomplished by use of an appropriate reagent which subjects the polished surface to chemical action. In alloys composed of two or more phases, the components are revealed during etching by preferential attack of one or more of these constituents by the reagent, because of the difference in chemical composition of phases.

In uniform single phase alloys or pure metals, contrast is obtained and the grain boundaries are made visible because of the differences in the rate at which various grains are attacked by the reagent. This difference in the rate of attack is mainly associated with the angle of different grain sections to the plane of polished surface. Because of the chemical attack by the etching reagent, the grain boundaries will appear as valleys in the polished surface.

Light from the microscope hitting the side of these valleys will be reflected out of the microscope, making the grain boundaries appear as dark lines.

The etching reagent used for Incoloy-800 is of the following composition.

| | | |
|---|---|---|
| Nitric acid | ($HNO_3$) | -40% |
| Water | ($H_2O$) | -30% |
| Hydrochloric acid | (Hcl) | -30% |

*E. Microstructure Analysis*

The microstructure of the specimens was analyzed using an Olympus optical microscope under the magnification of 100X.

The micrographs of these samples were taken and their grain sizes were measured using 'Arbitary Line Method', with the help of the software,' OLYSIA'. Arbitary Line Method: In this, a particular area of the specimen was focused.

The vertical and horizontal dimensions of all the grains present in the focused area were measured individually and their average was taken. The same procedure was repeated for three different areas of the same specimen and their average was considered as the average grain size of the specimen.

Fig. 9. Olympus optical microscope

## 5. Results and discussions

*A. Chemical Analysis*

Table 3

| Element | Wt.% of the element |
|---|---|
| Nickel | 30-35 % |
| Chromium | 19-23 % |
| Aluminum | 0.15-0.6 % |
| Carbon | 0.1 % max |
| Copper | 0.75 % max |
| Manganese | 1.5 % max |
| Silicon | 1 % max |
| Sulphur | 0.015 % max |
| Titanium | 0.15-0.6 % |
| Iron | Balance |

*B. Microstructure Analysis*

*1) Microstructure of Billet*

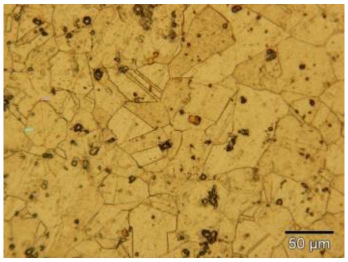



Fig. 10. Microstructures of the centre of the billet

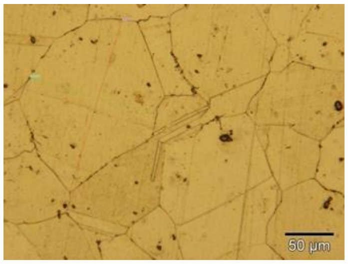



Fig. 11. Microstructures of the periphery of the billet

*2) Microstructure of the extruded tube of size 142mm dia x 50mm WT*

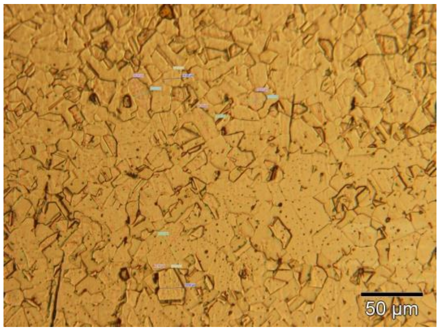



Fig. 12. Microstructure after extruded Longitudinal of the tube

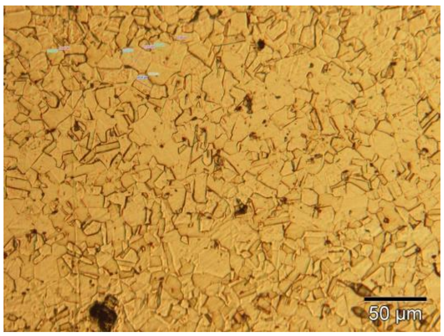



Fig. 13. Microstructure after extruded Transverse of the tube

*3) Microstructure of the pillgared tube of size OD 19.1 x 1.1 WT*

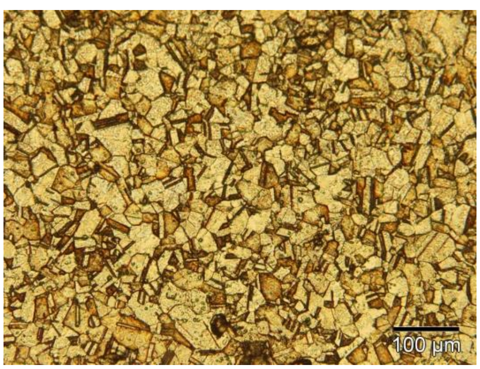



Fig. 14. Microstructure after final pillgaring inside tube


Fig. 15. Microstructure after final pillgaring outside tube

*4) Grain size*

- The average grain size of the billet at the centre in fig-16 has 2 microns grain size and periphery in the fig-17 has 4 microns grain size.
- The average grain size of the extruded tube at longitudinal in fig-18 has 4 microns grain size and transverse in fig-19 has 5 microns grain size.
- The average grain size of the final pillgared tube in fig-20 and 21 is 8 microns at longitudinal and transverse.

*C. Hardness values*

The hardness value of the Incoloy-800 is determined by Vickers hardness method. The Vickers hardness test method consists of indenting the test material with a diamond indenter, in the form of a right pyramid with a square base and an angle of 136 degrees between opposite faces subjected to a load of 30kgf.

Table 4

| Product | Hardness value (HV) |
|---|---|
| Billet | 140 HV |
| After extrusion of billet into tube | 160 HV |
| Final tube product | 290 HV |

The hardness value increases with decrease in the grain size. The hardness value of the billet is less than that of the tube after extrusion. The hardness value of the final tube product is high then that of other tubes.

*D. Strain Rate*

Calculating the strain rate of a billet having dimensions 1 mm OD x 45mm IDx 500mm length is extruded to 63mm OD and 9mm WT (main ram speed 100mm/sec)

We know $$€ = 6\,\frac{\text{x V ln(R) x } d^2 \text{ x tan}\alpha}{[D^3\text{-(Billet OD}^3)]}$$

Where
V=Main ram speed
Ln(R) =extrusion ratio (A/Af)
Tanα=semi cone diangle
D=outer dia of the extrusion product
D=outer dia of the billet

A = πt (OD-t) [A=initial thickness of the billet]
= 3.14(105/2) (150-150/2)
= 3.14 x 52.5 x 150-52.5
A = 16080

Af = πt (OD-t)
= 3.14 x 9 x 63-9
= 3.14 x 9 x 54
= 1526

A/Af=16080/1526=10.54 [A/Af=extrusion ratio]

Substituting extrusion ratio (Er) in strain rate

$$€ = \frac{1685 \text{ x } 600 \text{ x } 2035}{3124953}$$
$$= 0.73/\text{sec}$$

## 6. Conclusion

- The billet temperature should be maintained in between 1160°-1190°C.
- For 150mm OD x 45mm ID billet after extrusion tube size 63mm x 9mm WT the strain rate is 0.73m/s.
- The hardness value of the final tube product is high then that of the other tubes. The hardness value increases with decrease in grain size.

## References

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