Mechanical Properties and Microstructures of Incoloy 909 Rolled Rings Subjected to a Modified Forging Cycle.

A Senior Project

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Bachelor of Science

by

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Abstract

The microstructure of rolled rings of Incoloy 909 nickel-iron-cobalt superalloy subjected to a standard customer solution and aging treatment exhibited unacceptably large grains. Samples cut from a failed ring were mounted and polished for analysis. The samples were etched with a modified Kalling's waterless reagent and metallographic analysis was done to identify characteristic large grain structures distributed among the desired smaller grain structures. The large grains were identified and noted for comparison to later samples. Two test rings were forged out of Incoloy 909 using revised forging procedures involving reduced soak temperatures before each forging step. Metallographic samples were cut from micro-samples of each ring and mounted. The samples from the test rings were subjected to the same grinding, polishing, and etching procedure as the samples from the failed ring. Metallographic analysis was done on samples cut from the two test rings, which confirmed that the revised forging procedures had eliminated the large grain structures present in the failed ring, exhibiting instead a relatively uniform smaller grain structure. Tensile testing was done on samples from the test rings, reporting an average yield strength of 139.5 ksi for process A and 144.8 ksi for process B, an average ultimate tensile strength of 171.8 ksi for process A and 179.3 ksi for process B, and an elongation of 9.55% for process A and 10.5% for process B. These values were under the required minimum tensile properties of Incoloy 909 at room temperature, and the forging cycles were concluded to be unsuccessful.

Keywords: Materials Engineering, superalloy, Incoloy 909, forging, ring-rolling, tensile testing, metallography

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Approval Page

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Background

Incoloy 909 (Inco 909) is a nickel-iron-cobalt superalloy favored for its low thermal expansion and high strength retention at elevated temperatures. These properties make Inco 909 especially effective for use in jet engines combustion chambers and high temperature gas turbine casings (Figure 1). Nickel-based superalloys, such as Incoloy 903, are vulnerable to stress accelerated grain boundary oxidation (SAGBO), also called SAGBO embrittlement, causing failure of the component well before its expected lifetime[1]. SAGBO occurs due to oxygen exposure of the component at high temperatures while under stress, during which oxygen is charged into the grain boundaries. This embrittles the grain boundaries and allows for cracks to progress between grains, much like stress corrosion cracking. Since these alloys are often used, and indeed designed for, high temperature, high stress applications, SAGBO is highly detrimental. Inco 909, however, reduces the possibility of SAGBO with a 0.4 wt% silicon addition, while retaining the tensile and thermal properties of the material[2].

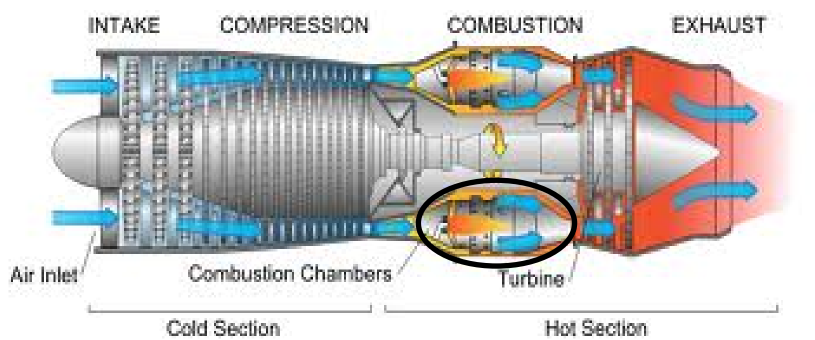

Figure 1: A cross section of a typical jet engine. Inco 909 is typically used in the combustion chamber, indicated[11]

This project is mainly concerned with the jet engine combustion chamber application of Inco 909, as the components studied were fabricated for this purpose. Ring-rolling is the preferred method of forging for combustion chamber applications and consists of several sizing and shaping processes with heat treatments in between each process, as discussed later.

Composition and Properties of Incoloy 909

Inco 909 is primarily a nickel-iron-cobalt alloy with some niobium and titanium for strengthening, and trace amounts of silicon, aluminum, and carbon (Table I). Niobium and

titanium decrease the thermal expansion coefficient and are constituents in the major strengthening precipitates. Unlike many nickel-based superalloys, Inco 909 does not contain chromium, as it drives the thermal expansion coefficient higher, thus compromising the effectiveness of the alloy in its preferred applications[3]. However, since Cr is not added, Inco 909 has a low corrosion resistance, meaning that it must be coated with a nobler material when put into service.

Table I: Composition of Inco 909 Superalloy[3]

Material	**Amount (wt%)**
Nickel	38
Iron	42
Cobalt	13
Niobium	4.7
Titanium	1.5
Silicon	0.4
Aluminum	0.03
Carbon	0.01

The properties of Inco 909 at room temperature are comparable to high strength steels, though the elongation of Inco 909 is significantly greater than most steels. At service temperatures, Inco 909 decreases in strength and increases in ductility as expected, though it retains enough strength to be acceptable in its applications (Table II). Minimum properties are important for designing components of jet engines and high temperature turbines, where in-service failures can be extremely dangerous.

Table II: Minimum Mechanical Properties of Inco 909 at Room Temperature and Service Temperature[3]

Temperature, °F	**Yield Strength, ksi**	**Tensile Strength, ksi**	**Elongation, %**	**Reduction of Area, %**
70	150	185	15	30
1200	125	150	25	60

Forging Processes of Metals

Metal forging is a group of processes in which a metal is subjected to stress to plastically deform it into a shape. Forging can take many forms, from closed die forging (Figure 2), where the metal is compressed between two molds that fit together, to open die forging (Figure 3), where the metal is formed between one or more mobile 'hammers', to rolling (Figure 4), where the metal is thinned by forcing it through two rolling cylinders. Forging can be done hot or cold, providing different properties of the material after forging. Inco 909 is typically forged using a type of rolling called ring-rolling. Ring-rolling is used to produce a ring geometry, such as that required for jet engine combustion chambers or high temperature gas turbine casings.

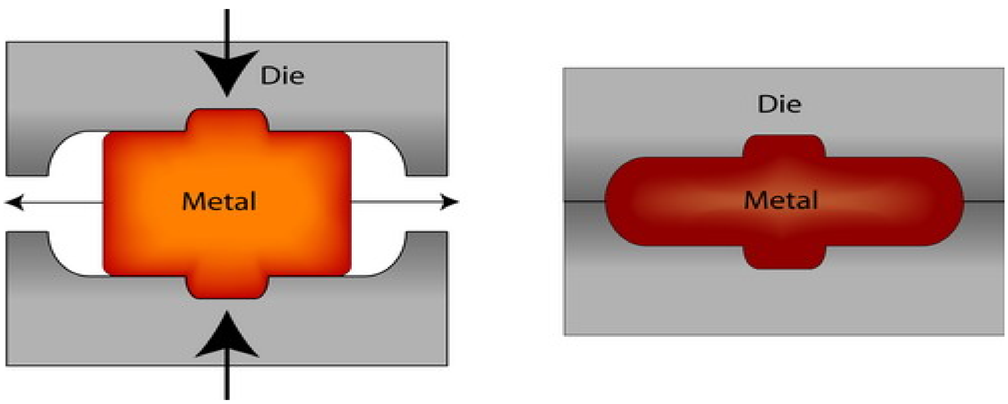

Figure 2: A schematic of a closed die forging process, used to form specific shapes[7]

Figure 3: An open die forge, used to form basic geometries in a workpiece[8]

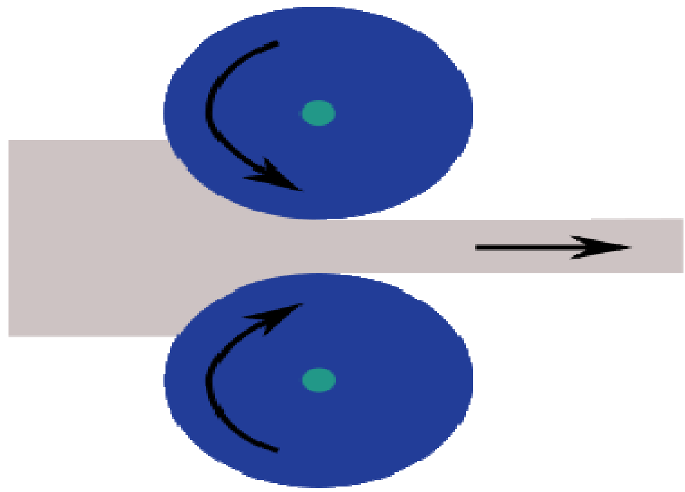

Figure 4: A schematic of rolling, used to reduce the thickness of the workpiece[12]

Ring-rolling begins with upsetting the stock material, usually a long cylinder, by compressing it between two flat dies to decrease height and increase diameter. The workpiece is then pierced by a flat cylindrical die pushed down the center to produce the hole in the middle of the ring. This ring is placed onto a mandrel and rolled in between the mandrel and a large cylindrical die, increasing the inner and outer diameters while reducing the thickness of the ring walls (Figure 5). In some cases, two other dies apply pressure to the faces of the workpiece to maintain a certain height (Figure 6). In the case of Inco 909, this process is performed at temperatures near 2000°F, with workpieces typically weighing around 200 pounds.

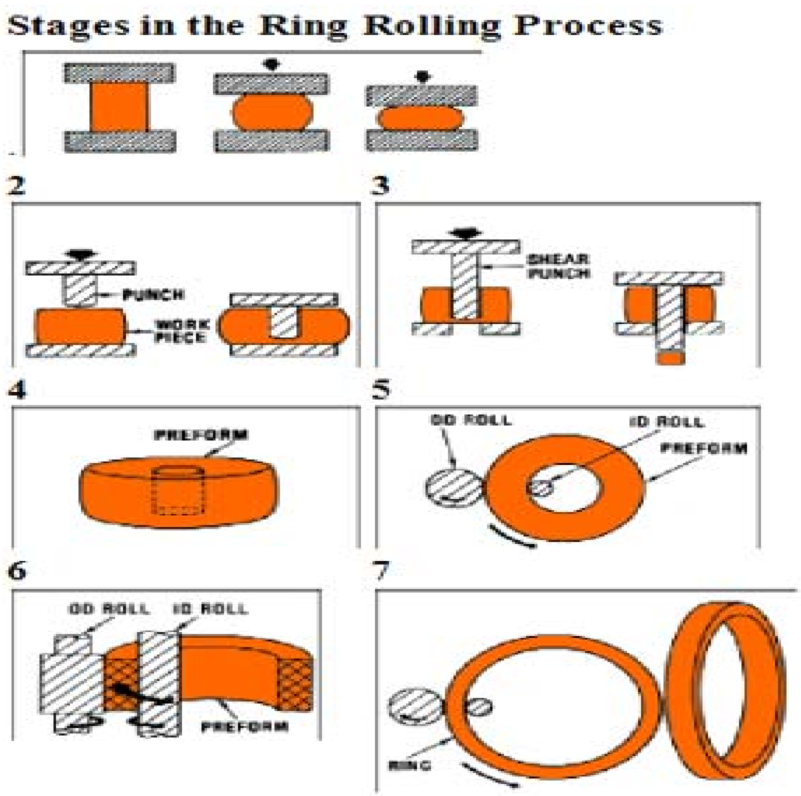

Figure 5: A schematic of a rolling process, illustrating the steps taken, resulting in the final ring geometry[9]

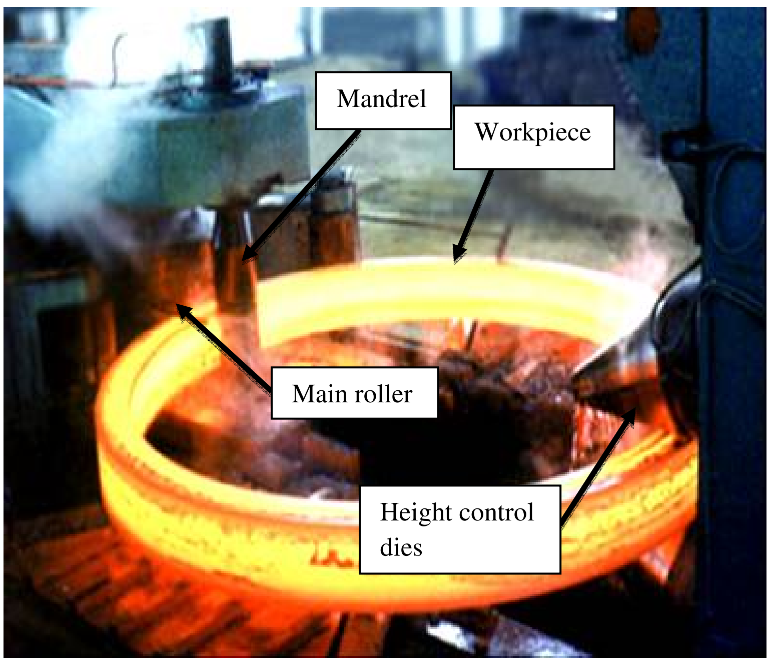

Figure 6: An example of ring-rolling in process. The main components have been labeled[10]

Problem Statement

Carlton Forge Works (CFW, Paramount, CA) was commissioned to fabricate ring forgings of Inco 909. After fabrication, it was observed that 'black grains,' or silicon rich grains, were present in the microstructure (Figure 7). These large, dark grains pose a problem for the strength of the alloy in service. The rings are required to have minimum safe tensile properties (Table II) and microstructures containing ASTM grain size 6 or finer. This project diagnoses the causes of the grain growth and investigates the effects of modifying the forging process has on grain growth and tensile properties. The focus of this project is on testing Inco 909 samples subjected to a new forging process for tensile strength and grain size.

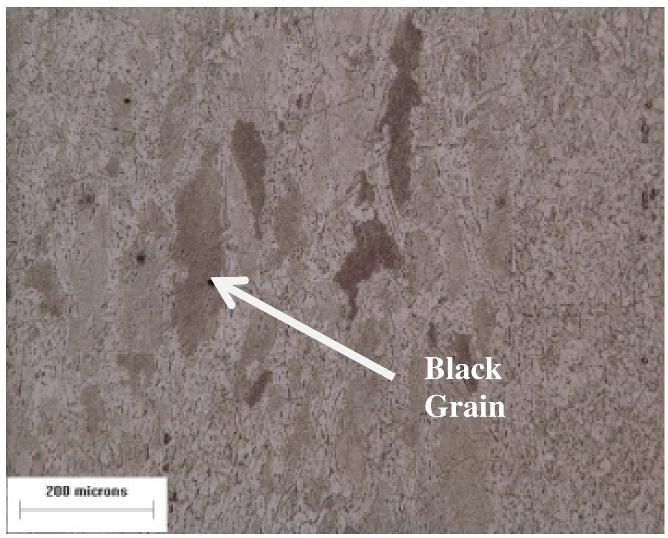

Figure 7: A micrograph at 100X, showing the 'Black Grains'

Experimental Procedure

CFW designed two modified forging cycles involving more rapid reductions of forging temperatures during the ring-rolling process. Two new processes were designed: process A, which involved a more gradual reduction of forging temperatures, and process B, which involved a more drastic reduction of forging temperatures (Figure 7). These changes were an effort to arrest grain growth during the forging cycle, since the heat treatment performed after forging could not be changed. A control group of conventionally forged Inco 909 samples was metallographically analyzed to provide an example of the 'black grains' to be compared with

later micrographs. The conventionally forged samples were processed at 2000°F for the first roll, and 1900°F for all subsequent rolls. All rings are air cooled to room temperature before and after the final heat treatment.

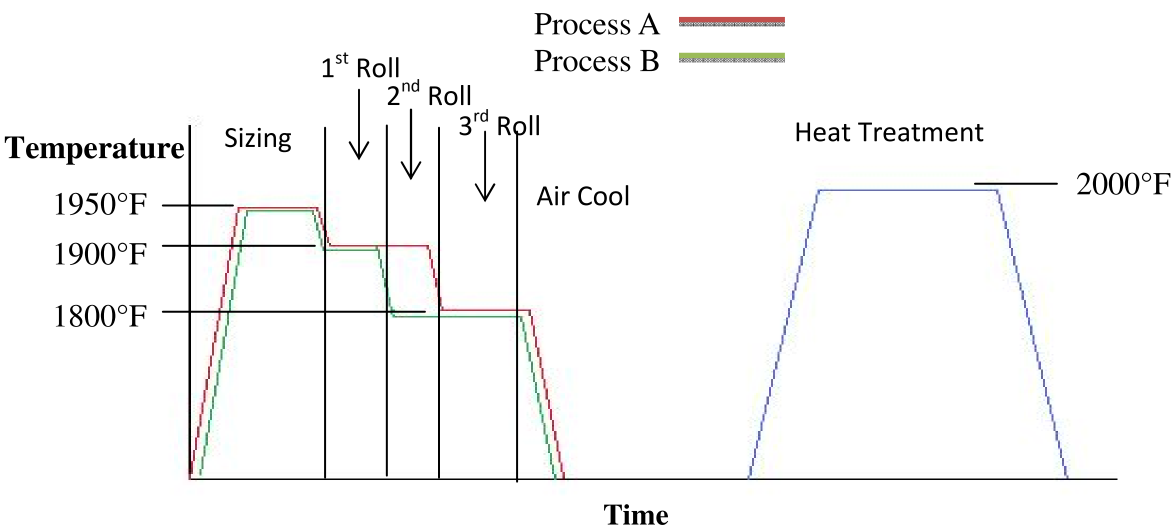

Figure 8: A representation of the modifications made in the forging temperatures

Inco 909 samples were machined from two rings, each forged under a different revised forging procedure. Five tensile samples were machined from each ring for the determination of the tensile mechanical properties of the rings. Metallographic samples were cut from each ring for metallographic analysis. Each tensile sample was tested to failure at room temperature (70°F) in an Instron tensile tester according to ASTM standard E8[5], and the tensile properties were recorded and averaged across a sample group. The metallographic sections were polished using 600 grit sandpaper, then 6 micron diamond polish and 1 micron diamond polish on low nap pads. The metallographic sections were etched with a modified Kalling's waterless reagent (100 mL methanol, 100 mL HCl, 5 g $CuCl_2$) to highlight the grain boundaries. ASTM standard E930-99 was used to find the size of the largest grain visible[6].

<u>Realistic Constraints</u>*:* Manufacturability and safety are the two main constraints involved in this project.

Manufacturability: In order to ensure that CFW is able to efficiently produce acceptable products of Inco 909, the manufacturability of the products must be considered. It is important that any solutions developed must be able to be put into application with a minimum of new equipment, a minimum of additional strain on current equipment, and a procedure that is indeed possible for CFW to carry out economically. Should the solution proposed require new equipment or put too much additional strain on current equipment, it will become too expensive for CFW to produce the products and may have to cease production of Inco 909 products altogether. The same ideas apply to the ease of implementation on the factory floor. Should a solution overcomplicate the processing steps, products may take too long to produce, or the margin for error may be too small, resulting in more rejected products than normal. Both would result in the possible ceasing of Inco 909 product production.

Safety: Of utmost importance is the safety of those forging the products and those who rely on the products in service with their lives. Serious injury and often death have resulted from failures in combustion chambers on jet engines. Minimum requirements must be met if there is to be a product at all. Also, the safety of those forging the products can be compromised if a solution applies techniques that the workers are unfamiliar with, or are just inherently dangerous, such as excessively high temperatures.

Results

Tensile mechanical properties measured at Cal Poly are presented in Table III. Tensile properties were also measured by a metallurgical testing company, Metals Technology, Incorporated (MTI Northridge, CA). MTI's results are presented in Table IV. The properties measured at Cal Poly did not meet the minimum mechanical requirements of Inco 909 alloy communicated in the literature. However, there was an issue with faulty calibration of the tensile tester at Cal Poly, which may invalidate these results. Results from MTI confirmed that the yield strengths did not meet the minimum requirements, but the ultimate tensile strengths and the elongation percentages did meet minimum requirements.

Table III: Tensile Test Result from Cal Poly

Process	Yield Strength, ksi	Tensile Strength, ksi	Elongation, %
A	146.90	180.50	14.50
	146.34	172.64	9.7
	144.60	178.98	8.3
	141.29	176.55	10.2
B	131.89	163.45	9.0
	139.50	172.64	9.70
	147.09	177.60	9.8
	139.70	173.65	9.7

Table IV: Tensile Test Result Averages from MTI

Process	Yield Strength, ksi	Tensile Strength, ksi	Elongation, %
A	148.20	186.20	14.5
	147.70	186.50	15.65
	148.60	185.30	14.65
B	149.50	189.00	14.75
	147.10	185.20	16.5
	149.40	189.30	14.65

Metallographic analysis yielded microstructures absent of ‘black grains’ present in the unmodified forgings, as well as exhibiting a certain amount of grain refinement (Figure 9). The grains exhibited straight, angular grain boundaries, suggesting that grain boundary pinning by precipitated lave phases occurred during forging (Figure 10). Twinning was also observed in the process B sample microstructure (Figure 11)

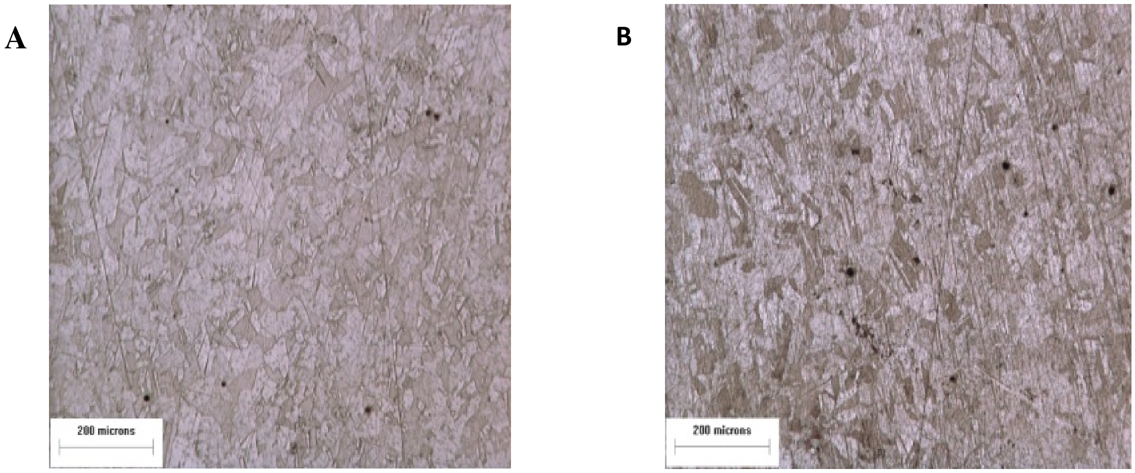

Figure 9: Micrographs at 100x of heat treated microstructures after forging. (A) Process A sample, (B) process B sample.

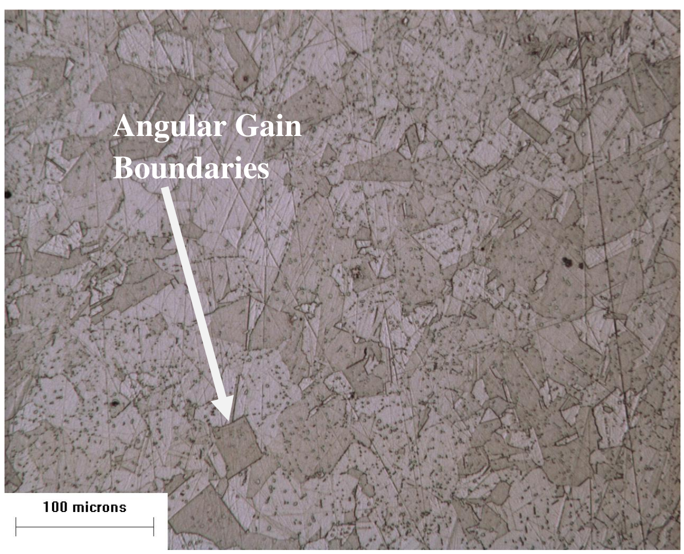

Figure 10: Micrograph at 200x of process A sample, illustrating the angular grain boundaries.

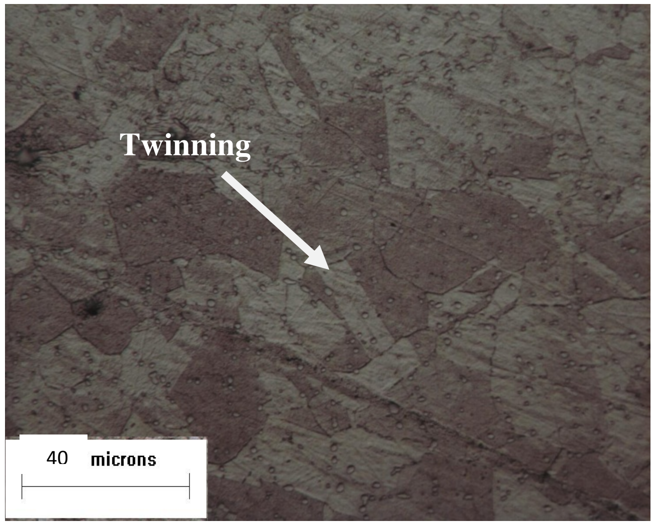

Figure 11: Micrograph at 500x of process B sample, illustrating the twinning within grains.

Discussion

The micrographs from samples from each forging process show that both forging cycles were successful in eliminating the 'black grains' from the microstructures and the grains were refined to the minimum size of ASTM 6. The absence of the 'black grains' confirms that their formation was due to overly high temperatures in the forging and heat treatment stages, which has been corrected by lowering the forging temperatures. However, the tensile properties acquired at Cal Poly did not meet the minimum requirements, namely the yield strengths. This suggests a problem with the formation of strengthening precipitates during both forging cycles and the required heat treatment. In Inco 909, grains are generally pinned during the initial high temperature forging cycle by the formation of multiple layer hexagonal laves in the microstructure, as mentioned in the results. The primary strengthening precipitates, γ' composed of $Ni_3(Nb, Ti)$, generally form at lower temperatures (Figure 12). Because the forging and heat treatments take place at temperature much higher than the temperatures in which the strengthening phases typically form, the high temperatures may be responsible for the decrease in tensile strength of the finished component. Also, heat treatment at such high temperatures may contribute to over-aging of any strengthening precipitates present in the microstructure. This may explain why the tensile properties are close to the minimum requirements, but are still not adequate.

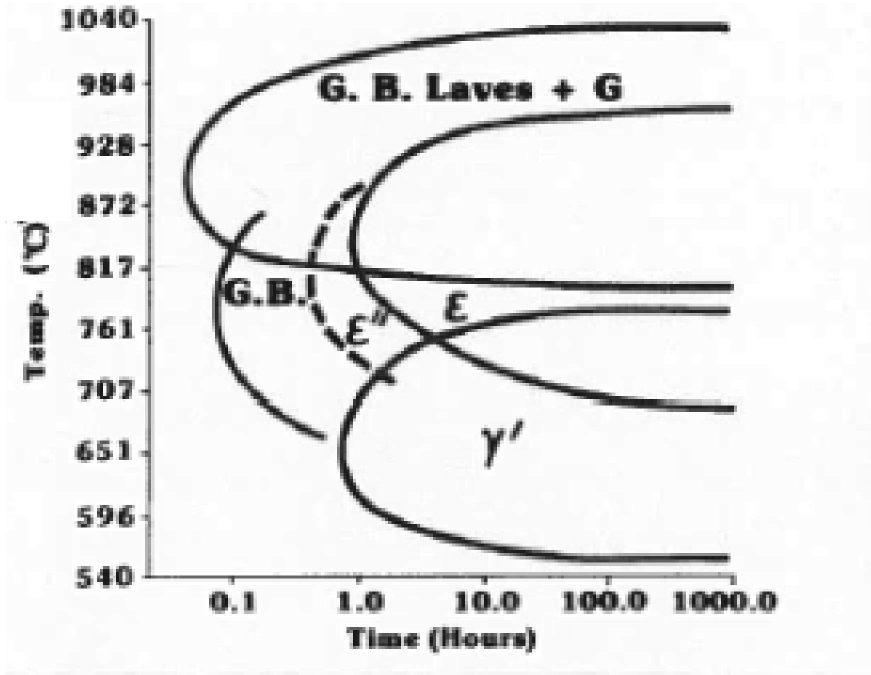

Figure 12: TTT plot for Incoloy 909 showing the areas of precipitate growth[2]

The results measured by MTI have acceptable elongation percentages and ultimate tensile strengths, but still show the yield strengths to be failing. These results may be indicative of the employment of one strengthening mechanism, that being the refinement of grains, and the lack of another strengthening mechanism, that being the formation of semi-coherent precipitates. Since the grains were refined from the failing microstructures, the greater density of grain boundaries would impede the motion of dislocations during plastic deformation, contributing to the ultimate tensile stress. If the strengthening precipitates were not formed in high enough concentration, the elastic deformation of the crystal structures would become less impeded, contributing to the reduced yield strengths.

Recommendations

In order to determine if the strengthening precipitates overaged or even formed at all, scanning electron microscopy (SEM) or transmission electron microscopy (TEM) imaging may be useful for determining problems with minimum mechanical property requirements. The forging procedure may need to be revised again, with forgings occurring at even lower temperatures to both arrest grain growth by pinning grains with precipitated lave phases, and promote the growth of peak-aged strengthening precipitates.

Conclusions

From the results, several conclusions can be drawn:

1. The modified forging procedures were successful in refining the grain structure and eliminating the undesirable ‘black grain’ from the microstructure, as per the original purpose of the project.
2. Due to heat treatment at exceptionally high temperatures, the precipitates usually responsible for strengthening the alloy may have had trouble forming, or were possibly overaged.
3. With consideration for the mechanical properties, the modified forging processes were unsuccessful in providing a viable option for an in-factory solution to the grain size problems.

Referneces

1. J. Rösler, S. Müller, "Protection of Ni-base superalloys against stress accelerated grain boundary oxidation (SAGBO) by grain boundary chemistry modification," *Scripta Materialia*, vol. 40, issue 2, pp. 257–263, 1998.
2. K. A. Heck, D. F. Smith, J. S. Smith, D. A. Wells, H. A. Holderby, "The physical metallurgy of a silicon-containing low expansion superalloy," in *Superalloys 1988,* S. Reichman, D.N. Duhl, G. Maurer, S. Antolovich, C. Lund, Ed. pp.151-160, 1988.
3. Special Metals Corporation, *Incoloy Alloy 909.* SMC-077, 2008, pp. 1-8.
4. McInnes Rolled Rings. (2013). "Seamless Rolled Ring Forging Process." *McInnes Rolled Rings.* Web. Available: http://www.mcinnesrolledrings.com.
5. ASTM Standard E8/E8M, 1999 (2011), "Standard Test Methods Tension Testing of Metallic Materials," ASTM International, West Conshohocken, PA, 2011.
6. ASTM Standard E930-99, 1999 (2007), "Standard Test Methods for Estimating the Largest Grain Observed in a Metallographic Section (ALA Grain Size)," ASTM International, West Conshohocken, PA, 2007.
7. "Closed Die." *Dissemination of IT for the Promotion of Materials Science, University of Cambridge* (2004): Web. Available: http://www.doitpoms.ac.uk/
8. S. Haverstock. "Breaking Corners on a Hot Billet." *New Opportunities with Open Die Forging* (2010): 24. Web. Available: http://www.gearsolutions.com/
9. "Stages in the Ring Rolling Process." *Monomet Incorporated*: Web. Available: http://www.monmet.com/
10. "D53-7000 cnc Radial-Axial Ring Rolling Mill." *TradeKorea.com* (2000): Web. Available: http://www.tradekorea.com/
11. J. Dahl. "Jet Engine." *Wikipedia.org* (2007): Web. Available: http://en.wikipedia.org/wiki/Jet_engine
12. Romary. "Laminage schema gene." *Wikipedia.org* (2007): Web. Available: http://en.wikipedia.org/wiki/Rolling_(metalworking)