EFFECT OF HEATING TEMPERATURE, HEATING TIME AND INITIAL MICROSTRUCTURE ON RECRYSTALLIZATION OF WASPALOY DURING HEATING FOR FORGING

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Approval Page

Project Title: Effect of Heating Temperature, Heating Time and Initial Microstructure on Recrystallization of Waspaloy During Heating for Forging

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Abstract

A fine, uniform grain size is required for creep limited Waspaloy in aircraft engine applications. Upon heating, Waspaloy does not always recrystallize in a uniform manner resulting in occasional large grains with a majority of the grains meeting the grain size requirements. Waspaloy's microstructure was analyzed with different heat treatments and in the as-forged condition. The strengthening mechanism is the gamma prime phase. The gamma prime solvus temperature is 1908°F so the different heat treatments were determined around that temperature, as well as the forging temperature range of 1800-2150°F. At the 1850°F heat treatment, the microstructure recrystallized with small grains, slightly larger than the as-forged samples. The samples heat treated at 1900°F had microstructures showing the sample recrystallized with larger grains surrounding the smaller grains with an ASTM grain size 5. The samples heat treated at 1925°F appeared similar to the samples that were heat treated at 1900°F, but had an ASTM grain size number slightly larger with an ASTM grain size number of 4.76. The samples heat treated at 1950°F showed larger grains than the previous heat treatments with an ASTM grain size 3.92. The samples heat treated at 2000°F showed much larger grains than the other heat treatments. The average grain size for the samples at 2000°F were about 2.4. The samples fell into the ASTM grain size number requirement for Schlosser at an ASTM grain size of 3 or finer, except for the heat treatments at 2000°F. The as-forged sample was approximately ASTM grain size 6. The recommended heat treatment temperature is 1925°F because it has the gamma prime strengthening mechanism and complies with Schlosser's ASTM grain size number specification requirements.

Keywords: Materials Engineering, Waspaloy, Forging Temperature, Recrystallization, Grain Size, Forging, Ring Rolling

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Introduction

Company Overview

Schlosser Forge Company (Rancho Cucamonga, CA) is one of the largest forging companies for the aerospace market. According to a recent news article, Pratt & Whitney awarded the Rancho Cucamonga-based Schlosser its Gold status for suppliers based on their on-time delivery, manufacturing quality, and customer service[1]. Their primary production is forged rings for aircraft engine applications. Their main method for processing is ring rolling. **Figure 1** shows a ring that Schlosser has ring rolled for their aero-bearing components[2]. The company works with many different superalloys, but the material of interest for this project is Waspaloy, a nickel-based superalloy.

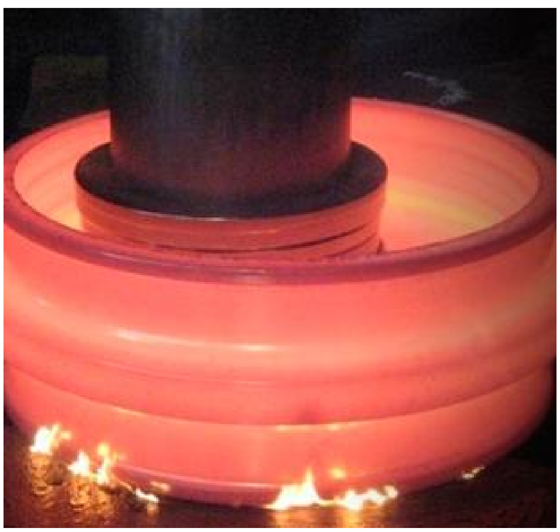


Figure 1: Ring that Schlosser has forged for their aero-bearing components[2].

Problem Statement

A fine, uniform grain size is required for creep limited Waspaloy. During reheating for forging, Waspaloy does not always recrystallize in a uniform manner resulting in occasional large grains. The majority of the grains meet grain size requirements, but occasional large grains can be seen (**Figure 2**). The creep that occurs that this elevated temperature causes microstructural changes that can degrade the material. This creates dislocations that are forced through the material resulting in work hardening[3]. There is currently not enough data to effectively select the optimal time and temperature combination to achieve a uniform recrystallized grain size distribution. The goal of this project is to determine the heating

temperature – time conditions that will result in a uniform grain size distribution without or at least with fewer occasional large grains.

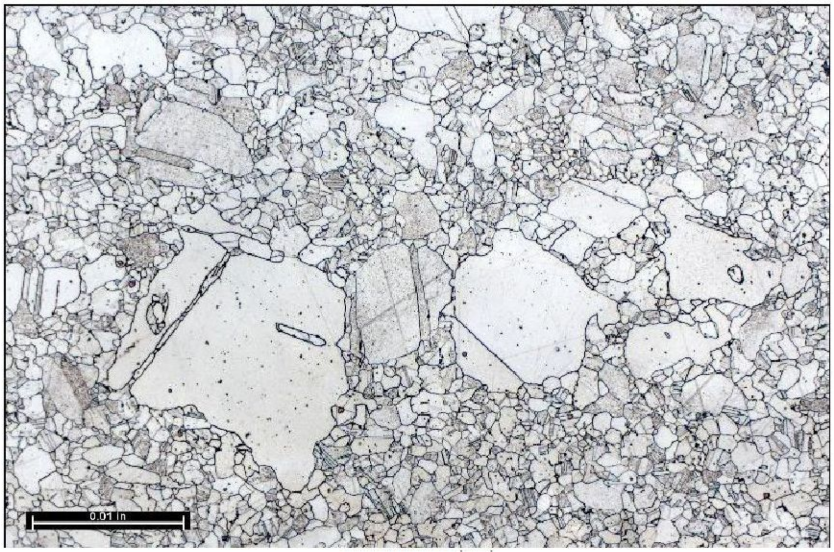


Figure 2: Waspaloy microstructure containing as-large-as (ALA) grains[4]

Nickel-based Alloy

Nickel-based alloy billets can be induction heated or furnace heated prior to hot forging. For this study, furnace heating will be employed for the heating of the billets. Metals that have been exposed to sulfur at elevated temperatures usually are mechanically weakened along the grain boundaries. This will cause the material to fracture during the forging process. There should be a fuel to air ratio with more fuel then air. High amounts of carbon monoxide or free carbon are not harmful since nickel-based alloys will not carburize at these conditions[5].

Superalloys

Superalloys are alloys that are used at high temperatures because of their ability to maintain strength at temperatures up to 0.8 the melting temperature. Superalloys are also used because of their creep resistance, corrosion resistance, and their high-cycle fatigue strength. For this project, Waspaloy is the superalloy of interest. Waspaloy is a nickel-based superalloy with a chemical composition of Ni-19.5Cr-13.5Co-4.3Mo-3Ti-1.4Al-<2.0Fe-0.07C[6]. Waspaloy's strength control comes from the gamma-prime phase (Ni_3 (Al, Ti)).

Waspaloy's strengthening comes from the gamma prime phase resulting in precipitation hardening. Most of that strength depends mainly on aluminum and titanium to allow precipitation hardening to occur. Precipitates strengthen Waspaloy by impeding the deformation

process. The deformation process can be impeded in three ways[7]. The first way is the degree of mismatch between the matrix and the precipitates. The second way is the order of the precipitates. The third way is the size of the precipitates.

Gamma prime gets its strength from the coherency strain within the lattice. The lattice mismatch between the precipitates, γ', and the matrix, γ, causes this coherency strain[8]. Waspaloy has a gamma prime volume fraction of about 30%[9]. The interface coherency is when the planes and directions line up in the matrix and precipitates. During coherency, dislocations can still move, but it is hard for them. The strength when the precipitates are small comes from the strain energy. As the precipitates get larger, the strength starts to come from the surface energy. Some properties of Waspaloy can be seen in **Table I**. Strain non-uniformity can also occur during forging when there is not a uniform temperature throughout the component[5]. This is because the component has different viewpoints in respect to the heat source from the furnace depending on the geometry so it is important to control uniformity.

Table I: Properties of Waspaloy[10]

Young's Modulus	USD/lbs	Forging temp	Density 1b/in^3	Heat capacity (BTU/lb.°F)	Tensile Strength	Yield Strength
30.6-32.2 x10^6 psi	20.50-22.50	1800-2150°F	0.297-.299	0.123-0.128	160-177 ksi	110-125 ksi

Along with controlling uniformity of the grain sizes, it is important to minimize creep deformation at elevated temperatures. Creep can be described as time dependent, temperature accelerated plastic deformation. Creep test are usually done at constant temperatures and stress. Many engineering alloys show a steady-state strain rate behavior for certain temperature-stress combination. The steady-state creep rate usually depends on the stress applied. Dislocation and diffusion creep can control slow deformation of materials at elevated temperatures[11]. High temperature creep typically occurs at the grain boundaries. In Ni-based superalloys, creep resistance is attributed to a substantial amount of γ' phase hardening due to the addition of Al and Ti.

Forging Process

Forging is the metal working process used to make the rings that Schlosser produces for turbine casing and turbine spacers. The forging process used is ring rolling. For their specific process, they start with a cylindrical Waspaloy billet that is ten inches in diameter. When the billet is ready to undergo the forging process, it is heated in a high temperature furnace. The furnace heats the billet until the forging temperature is reached. The billet is then placed between a hydraulic press. The hydraulic presses used at Schlosser ranges from 750-4000 tons. The hydraulic press flattens the billet and the center is then punched out. Once the center is punched out, the billet is placed in a ring rolling mill. There are two types of ring rolling mills: vertical and radial axial. Radial axial ring rolling machines are the ring rolling mills used. **Figure 3** shows a billet being rolled using radial axial ring rolling.

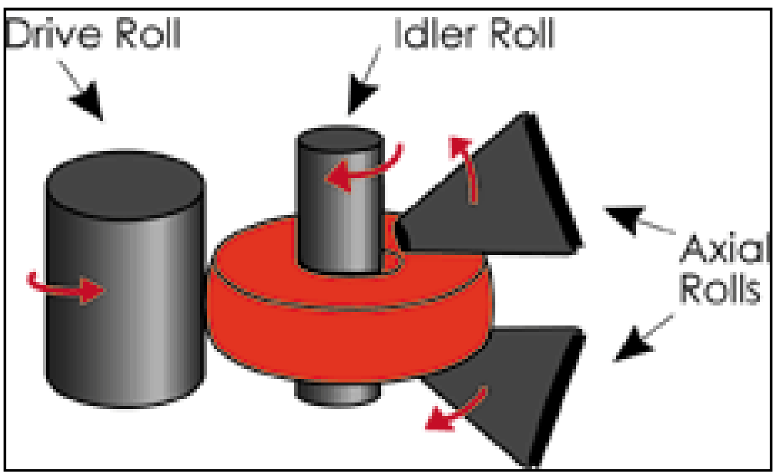


Figure 3: Radial axial ring rolling with two-passes[12].

Figure 3 shows two passes which means there are two axial rolls. The two axial rolls force the reduction of the height of the ring. The axial rolls turn away from each other; this torque causes the ring to rotate[5]. The drive roll also rotates and restricts the wall thickness to obtain the desired diameter. **Figure 4** illustrates how the ring looks in the midst of ring rolling process.

Figure 4: Ring rolling to a large diameter[13].

Realistic Constraints[14]

The realistic constraints that ABET states for their criteria for accrediting engineering programs for the student outcome consists of many criteria. The criteria are:

(a.) an ability to apply knowledge of mathematics, science, and engineering

(b) an ability to design and conduct experiments, as well as to analyze and interpret data

(c) an ability to design a system, component, or process to meet desired needs within realistic constraints such as economic, environmental, social, political, ethical, health and safety, manufacturability, and sustainability

(d) an ability to function on multidisciplinary teams

(e) an ability to identify, formulate, and solve engineering problems

(f) an understanding of professional and ethical responsibility

(g) an ability to communicate effectively

(h) the broad education necessary to understand the impact of engineering solutions in a global, economic, environmental, and societal context

(i) a recognition of the need for, and an ability to engage in life-long learning

(j) a knowledge of contemporary issues

(k) an ability to use the techniques, skills, and modern engineering tools necessary for engineering practice.

This study will impact the manufacturability of Waspaloy and the economy. This study will provide a better understanding of the temperature and time combination that Waspaloy should be reheated to between the forging steps to minimize the growth of occasional large grains within the microstructures. This will make Waspaloy easier to manufacture for turbine casing and turbine spacers because there will be a fine, uniform grain structure that will be less likely to crack during the forging process.

The economy will be influenced because Schlosser will no longer have to be concerned about the temperature at which these occasionally large grains occur so they will no longer have to waste energy by maintaining the furnaces at higher temperatures. They will also not have to waste material because it did not pass their ultrasonic testing requirements for the microstructure. The ultrasonic inspection consists of beam scan which detects the defects in inches and the percent rejection. The scanning speed is 70 surface feet per minute.

Experimental Procedure

Waspaloy's recrystallization was analyzed in the as-forged samples and at different temperatures and times. The heat treatment matrix can be seen in **Table II** with corresponding sample labels. The heat treatment temperatures were chosen because the forging temperature range is 1800-2150°F and the gamma prime solvus temperature is 1908°F. The two outer temperatures were chosen in order to have a reference point for the microstructures. The times were determined in order to provide a range of length the forging components will be in the furnace for reheating. Each sample was taken out at the appropriate times and water quenched. Once the heat treatment was completed, the samples were prepared for metallography.

Table II: The Heat Treatment Matrix of Waspaloy Samples

Temperature	One Hour	Two Hours	Four Hours
1850°F	B1	-	B4
1900°F	C1	C2	C4
1925°F	D1	D2	D4
1950°F	E1	E2	E4
2000°F	F1	-	F4

Each heat treatment sample was sectioned and mounted in mineral-filled diallye phthalate. They were grinded through the 240, 320, 400, and 600 grit SiC papers. They were than polished with the six and one micron polishing pads. The samples were etched with waterless Kalling's reagent which consists of 2g $CuCl_2$, 40 mL HCl, and 80 mL methanol[15]. Since the Metals Handbook said the samples could be swabbed or immersed, both methods were attempted. The samples with lower heat treatment temperatures were etched by swabbing the samples. The samples that had higher heat treatment temperatures were etched by immersing the samples in a beaker that contained enough of the etchant to coat the surface of the sample. Two etching methods were used because the samples that had heat treatments with lower temperatures appeared to etch faster than the samples with higher heat treatment temperatures.

Once the samples were etched to the point that the grain boundaries were visible, a software program (IQmaterial) was used to determine the ASTM grain size number. The micrograph of the sample was placed in this program and points were made where the program's reference lines intersected the grain boundaries. The manual markings were counted up and then were divided by the length of the reference lines. This was done at five separate locations for a given sample. These five numbers were averaged to give one *n*, where *n* is the number of grains per square mm. It was then used in **Equation 1** to find the ASTM grain size number[16].

$$N = \frac{\log n}{\log 2} + 1.000 \quad (1)$$

After the ASTM grain size number was determined, the values were analyzed. The grain sizes were averaged for each of the temperatures since time did not appear to influence the grain size. The larger the ASTM grain size number, the finer the grains are within the microstructure. Schlosser's specification requirements were an ASTM grain size 3 or finer.

Results

From the micrographs observed, the ASTM grain size number was determined. The samples that were as-forged had an ASTM grain size of 6.21 and 6.39. These were the finest grains that were analyzed in this experiment. **Figure 5** shows the average ASTM grain size number of the samples heat treated. As the heat treatment temperature increases, the ASTM grain size decreases which means the average grain size is increasing. The specification requirements are for an ASTM grain size 3 or finer. Figure 5 also illustrates that only one of the heat treatments does not meet the required ASTM grain size number specification, 2000°F.

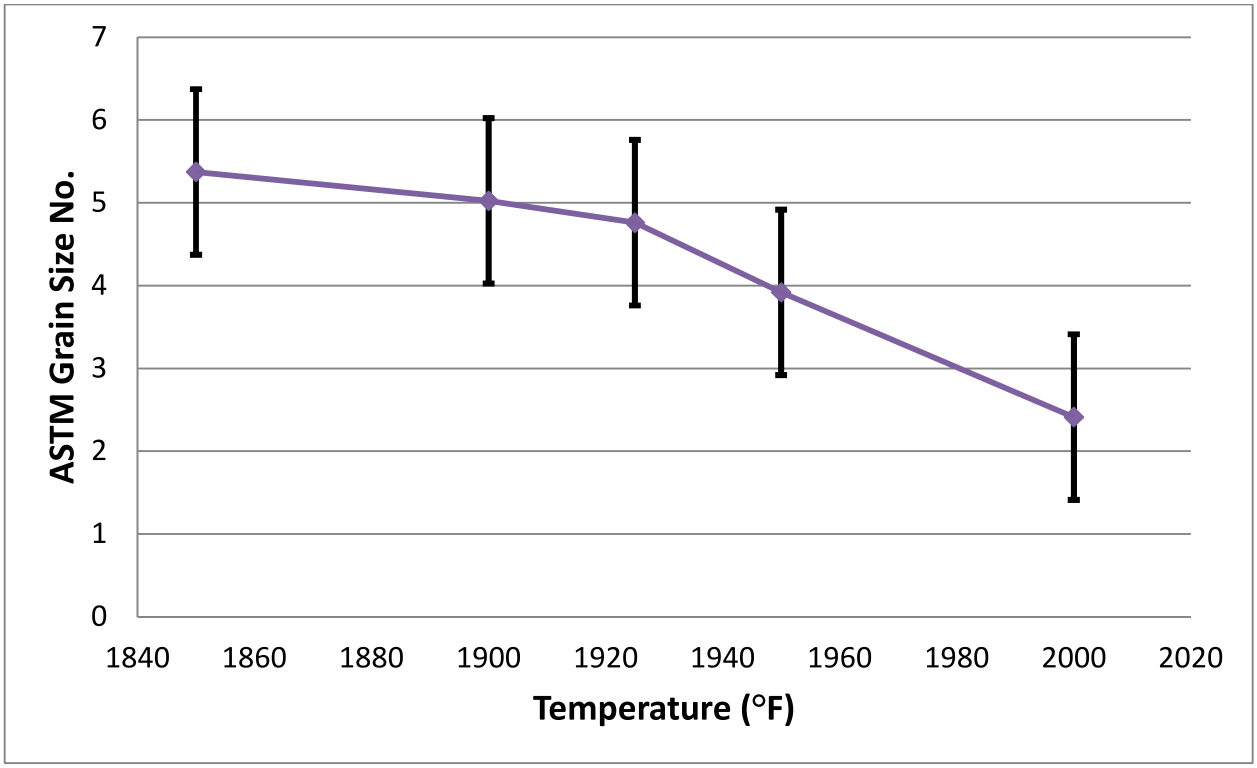


Figure 5: The average ASTM grain size number for Waspaloy at different heat treatment temperatures with a standard deviation of 1.

The samples were averaged for each heat treatment temperature because the samples did not vary based on the time in the furnace, but only based on the different heat treatment temperatures (**Table III**). The two heat treatments that are below the gamma prime solvus temperature have similar ASTM grain size numbers. This could be due to the precipitation hardening from the gamma prime phase occurring higher than 1900°F.

Table II: The ASTM grain size number for Waspaloy

Temperature (°F)	One hour	Two hours	Four hours
As-Forged	6.3		
1850	5.55		5.19
1900	4.95	5.05	5.07
1925	4.74	4.77	4.77
1950	3.98	3.59	4.19
2000	2.39		2.43

The measured ASTM grain size number decreases with a temperature increase, but is not based on the time; this could be because there is enough time for recrystallization to occur. However, there is not enough time for much grain growth. The samples recrystallize to align themselves in a lower strain energy state. To better understand how the sizes of grains change during the heat treatments, one can observe **Figure 6** to see the average grain sizes in microns.

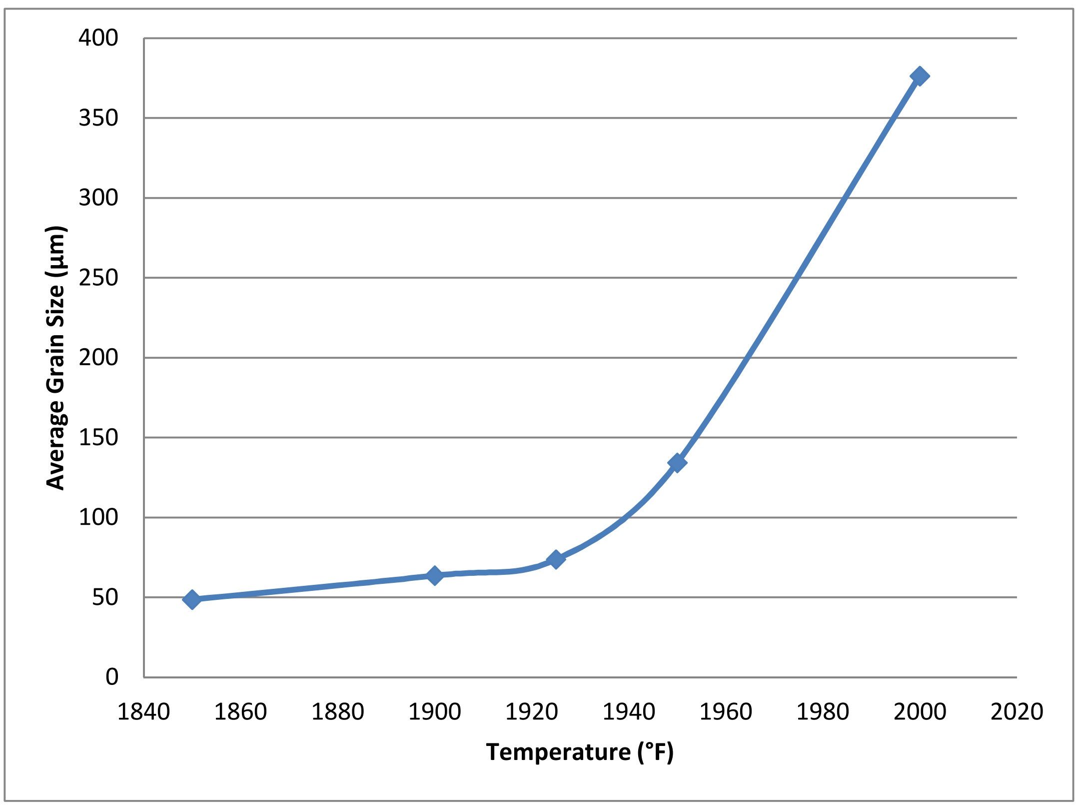


Figure 6: The average grain size (in microns) of Waspaloy at each heat treatment temperature.

The curve of the average grain size shows a drastic increase of grain size as it goes from a heat treatment temperature of 1950°F to a temperature of 2000°F. The temperatures 1850 and 1900°F are 50 degrees apart, but the samples at those temperatures do not show a drastic difference in the grain sizes because they are both below the gamma prime solvus temperature (1908°F).

The ASTM grain size number decrease as the heat treatment temperature increased. The as-forged samples showed fine grains (**Figure 7**) that met the specification requirements. As the temperature increased, the recrystallized grain size increased. The samples slowly started to form the occasionally large grains that were surrounded by smaller grains as the temperature increased. The time for heat treating did not appear to influence the grain size for a given heat treatment temperature which is evident since the ASTM grain size number stayed the same for each time interval for a given heat treatment temperature.

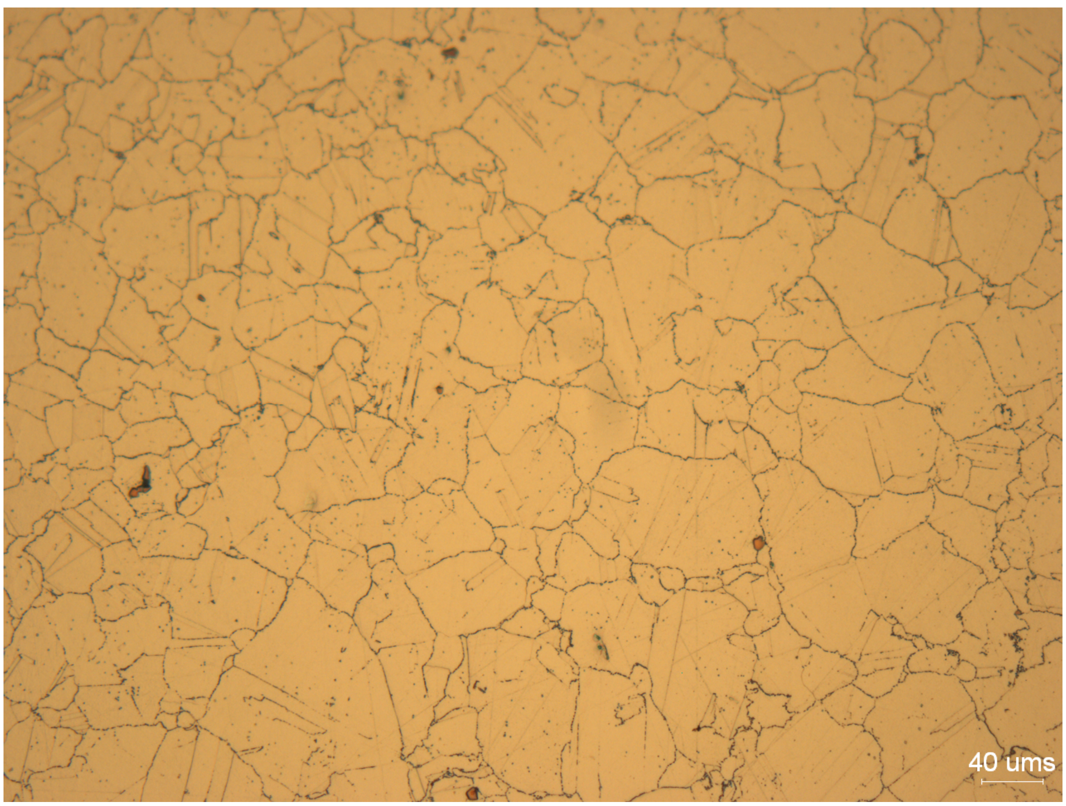


Figure 7: Fine grains of the as-forged Waspaloy at 200x are shown with an average ASTM grain size of 6. This sample was etched with waterless Kalling's reagent.

The as-forged samples were measured to have an average ASTM grain size of 6.3. This sample was the only sample that was not heat treated. The first sample that was heat treated is shown is shown in **Figure 8**. This sample was heat treated at1850°F. The microstructure at 1850°F does not suggest it will cause failure due to irregular large size grains. This microstructure clearly shows that the sample recrystallized in a uniform manner which could be attributed to this heat treatment temperature being below the γ' solvus temperature.

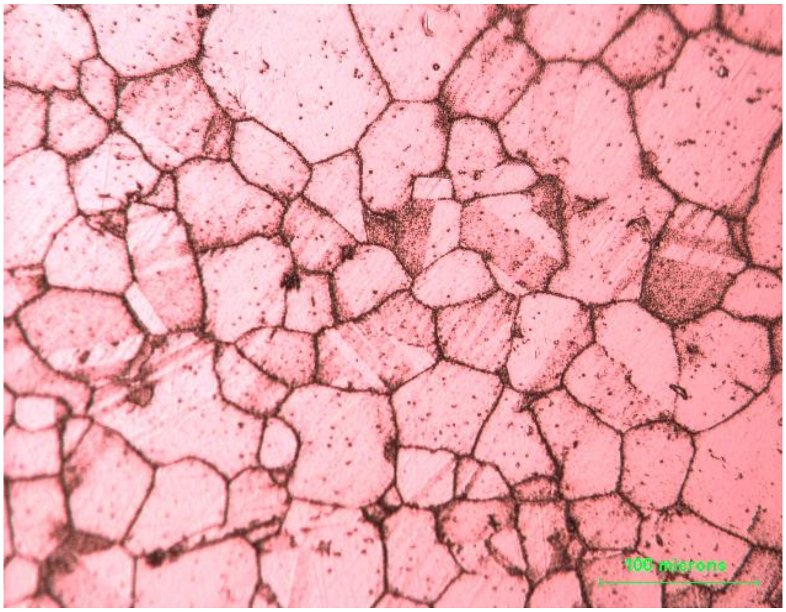


Figure 8: Waspaloy at 1850°F heat treatment for four hours. This sample was etched with waterless Kalling's reagent and is shown at 200x.

The microstructures of the samples at 1900°F show obvious signs of larger grains growing. This can be seen in **Figure 9**. This sample is slightly lower than the γ' solvus temperature which is 1908°F. This is the first sample that starts to show the occurrence of occasional large grains. In this sample, one can see the twins from annealing[8].

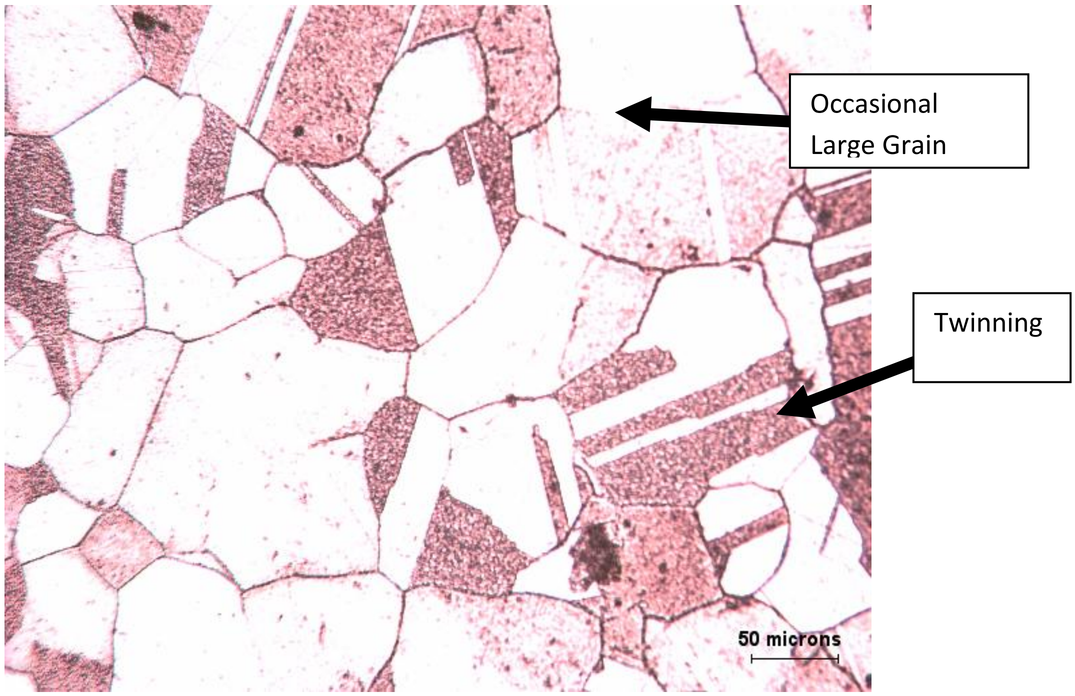


Figure 9: Waspaloy annealed at 1900°F for one hour at a magnification of 200x. This sample was etched with waterless Kalling's reagent.

The heat treatments at the same temperature, but different times show similar ASTM grain sizes which can be seen in **Figure 10**. The samples at 1900°F show the development of the occasionally large grain sizes. This is attributed to the fact that the heat treatment is so close to the gamma prime solvus temperature.

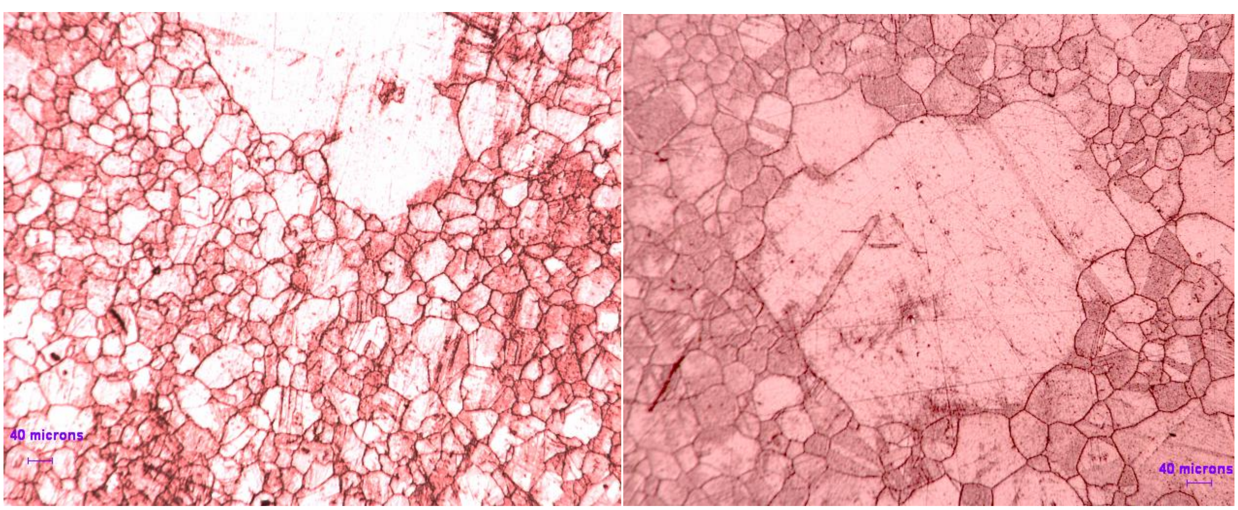


Figure 10: Waspaloy heat treated at 1900°F for two hours (left) and at four hours (right) at 100x. These two samples contain the occasional large grains within the microstructure.

The samples at 1925°F noticeably follow the pattern of increasing the grain size of the occasionally large grains (**Figure 11**). This is the first heat treatment that was done above the gamma prime solvus temperature. Since this heat treatment is above the gamma prime solvus temperature, the precipitates forming results in a mismatch lattice of the precipitates and the matrix. This sample is stronger than the samples below the gamma prime solvus temperature, but still contains the occasional large grains. This gamma prime solvus temperature is the temperature where precipitation hardening starts to occur. It is stronger because it is higher than the previous heat treatment temperatures so there are more precipitates that strengthen the microstructure

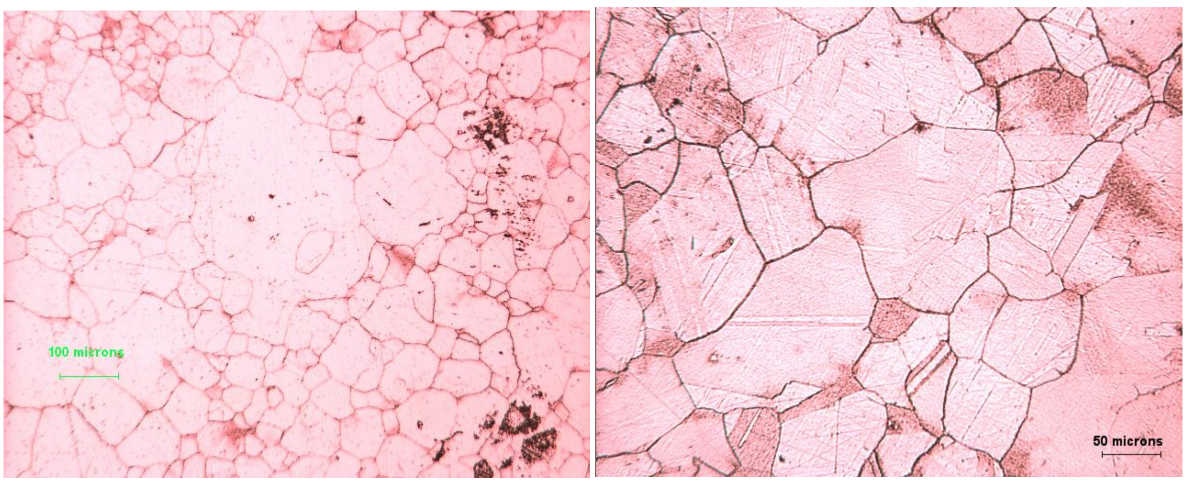
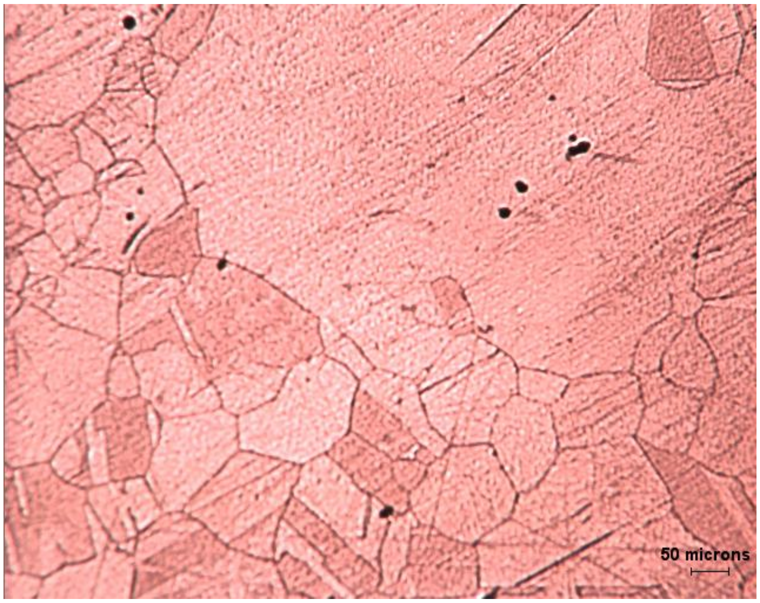


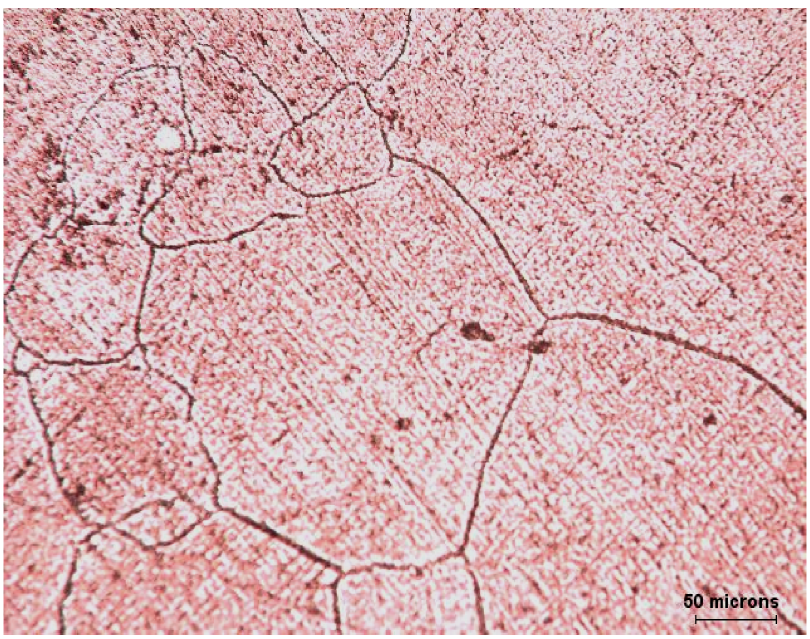
Figure 11: Waspaloy sample heat treated at 1925°F for one hour (left) and for four hours (right) at 100x.

Thus far, there is an obvious trend that shows that at elevated temperatures the formation of occasionally large grains in the microstructure increases in size, not necessarily the number of large grains present (**Figure 12**). Large grains usually form when the rate of nuclei is small and the rate of growth is larger. The recrystallization of these grains also deals with the percent of work that was done on the material. The material with the largest amount of work done on it will have smaller recrystallized grain size. A low percentage of work done on a material will result in larger recrystallized grain size.



’F at 100x. This sample
nt.

The last heat treatment done was conducted at 2000°F (**Figure 13**). This heat treatment was done on the outer limits of Waspaloy’s forging temperature range (1800-2150°F). As predicted, the sample heat treated at 2000°F resulted in large grains within the microstructure. These grains appeared to be larger than the previous microstructures. This sample is about 100°F above the gamma prime solvus temperature.



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The recommended heat treatment would be at 1925°F because it is above the gamma prime solvus temperature so the material will have smaller size of occasional large grains compared to the higher heat treatment temperature. Even though the occasional large grains still occur at 1925°F, any lower heat treatment temperature will reduce the strength because it will be below the gamma prime solvus temperature.

Discussion

At each heat treatment, the samples passed the ASTM number requirement except for the samples that were heat treated at 2000°F. Although most of the samples passed the grain size specifications, it is clear that there are occasional large grains present within the microstructure starting at the 1900°F heat treatment. This could be influenced by the method used when measuring the grain size of a sample.

The measuring method involves a reference image used to mark where the grain boundaries intersect with the reference image. This means that the reference image may only contain a small part of a large grain while intersecting various parts of the small grains. More intersections on the reference image will occur with smaller grains because it depends on how many of the large grains are measured for the given samples. When the markers are averaged for the five different locations on the sample, the large grains do not influence the grain size number as much as the smaller grains. As the large grains grow bigger with increasing the heat treatment temperature, they begin to influence the average grain size more because they take up more space on the reference image. The cause of these occasional large grains is not determined yet, but there is a theory. In the Materials Science Forum Journal, J. Dennis said, "Minority texture components may experience accelerated growth due to a higher energy and mobility compared to the surrounding grain structure. The combination of these two events may result in the abnormal growth of some grains[17]."

The grain size only dealt with the heat treatment temperature and not the time for each heat treatment. The recrystallization temperatures must be below 1850°F because there is a difference between the as-forged and the first heat treatment where the ASTM grain size number went from a six to about a five. The grain size is also influenced by the amount of work done to the samples during forging. Since the work applied to the samples was not uniform throughout the part, there will be different amounts of work done at different sections of the ring. The sections of the ring with less work done will have a larger recrystallized grain size than the sections with more work which could be the reason for the occasional large grains. The sections that have more work applied to it will have more defects, meaning more grain boundaries.

Conclusions

1. The recommended temperature for forging Waspaloy for Schlosser's turbine spacers and turbine casing would be 1925°F because it is above the gamma prime solvus temperature and still passes the ASTM grain size number specification by having an ASTM grain size number of 4.77 which is a finer grain size number than the specification required.
2. Although the samples at 1925°F show occasional large grains, it has the strength from precipitation hardening as well as meeting the requirements since it is above the gamma prime solvus temperature.
3. The samples at 1900°F meet the grain size requirements as well, but are below the gamma prime solvus temperature which means they are not employing Waspaloy's strengthening mechanism of the gamma prime phase.
4. Time does not influence the grain size for a given heat treatment.

References

1. Pierceall, Kimberly. "AWARD: Schlosser Forge Wins Gold Honor." *Press-Enterprise* 24 Jan. 2012.
2. Aero-bearing Component. Schlosser Forge Co. May 2012. <http://www.firthrixson.com/aero-bearing>.
3. ASM Handbook. Volume 11, Failure Analysis and Prevention. Creep and Stress Rupture Failures: Bulk Creep Behavior. 2003.
4. Waspaloy microstructure, Schlosser Forge Co. Personal photograph by author. 2012.
5. Semiatin, S. L. *Metalworking: Bulk Forming*. Vol. 14A. Materials, Park, Ohio: ASM International, 2005.
6. Material Background. 2012. Seco Tools. May 2012. <http://www.secotools.com/sv/Global/Segment-Solutions/Aerospace-Solutions/AS-Material-main/Heat-resistant-super-alloys/Inconel-71871/>.
7. Donachie, M.J., and S.J. Donachie. "Understanding Superalloy Metallurgy". Superalloys: A Technical Guide. 2nd ed. 2002. ASM Handbook Supplements, 2011.
8. Abbaschian, Reza, L. Abbaschian, and R. Reed-Hill. *Physical Metallurgy Principles*. 4th Ed. Stamford, CT: Cengage Learning, 1994.
9. Guttmann, V. *Phase Stability in High Temperature Alloys*. London: Applied Science, 1981.
10. Materials Universe: Metals and alloys\Non-ferrous\Nickel\Chromium alloy\Wrought\Waspaloy. CD-ROM. CES Edupack 2011.

11. Bressers, J. *Creep and Fatigue in High Temperature Alloys*. London: Applied Science, 1981.

12. Radial Axial Ring Rolling with two-passes. Jan. 2012. <http://www.scotforge.com/images/photos/rprocess4.gif>.

13. Ring Rolling to a larger diameter. Jan. 2012. <http://img.tootoo.com/mytootoo/upload/89/890801/product/890801_d0f6d561554aee74f4c64111eaf55149.jpg>.

14. ABET Criteria for Accrediting Engineering Programs 2010-2013. General Criteria 3: Student Outcomes.

15. Metals Handbook, Ninth Edition. Vol. 9: Metallography and Microstructures. American Society for Metals. Ohio, 1985. Pg. 308.

16. Metals Handbook, Ninth Edition. Vol. 9: Metallography and Microstructures. American Society for Metals. Ohio, 1985. Pg. 129.

17. Dennis, J., et al. Materials Science Forum Journal 2007: 558-559, 717.