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METALLURGICAL CHARACTERIZATION OF WASPALOY PRESENTING VARIATIONS IN CHEMICAL COMPOSITION, GRAIN SIZE AND HARDNESS

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Abstract

Waspaloy is a superalloy used to manufacture some aeronautical parts subjected to high temperatures and stresses. During thermo-mechanical processing cracks are generated, causing some parts to be rejected. In order to determine the causes of these cracks, this alloy was characterized using techniques such as chemical analysis, optical microscopy, scanning electron microscopy, and EDX analysis. Heterogeneous grain sizes in the microstructure cause a non-uniform strain distribution in these parts, creating cracks in zones with different grain sizes.

Introduction

Nickel-base superalloys are important materials for high-temperature service applications. The manufacture of components from these materials is typically based on solidification or powder-metallurgy processes [1]. Solidification techniques comprise either the direct casting of parts or the casting of ingots which subsequently undergo a series of thermo-mechanical-processing steps to refine the microstructure. During solidification, microsegregation is usually unavoidable. The primary solidification product is a disordered nickel solid solution with an fcc crystal structure (γ). In many alloys, the material which solidifies last is a mixture of γ and an ordered fcc phase (γ'). In prototypical nickel-base superalloys, the γ'-phase is $Ni_3(Al,Ti)$ [2-3].

Waspaloy is a superalloy that is used in demanding high-temperature environments in which good creep and fatigue resistance are required. One of the most common applications of Waspaloy is for turbine engine rings. To ensure the required mechanical properties, the rings are usually forged and heat treated (Figure 1). Two distinct types of microstructures are usually found to be attractive for such applications: a) A microstructure with a grain size of ASTM 10 to 14 for tensile strength, ductility, and resistance to crack nucleation in low-cycle fatigue; or b) A microstructure with a grain size of ASTM 4 to 8 required for creep strength and resistance to crack propagation [4]. Because it is impossible to refine the grain size through heat treatment, a skillfully designed forging process is crucial for the control of the grain size and, hence the properties of the forged ring [5-6].

To obtain optimum properties, γ´-strengthened superalloys must be homogenized following casting (or prior to hot working in ingot-metallurgy approaches) at temperatures between the γ´-solvus and the solidus. At these temperatures, the γ´-phase dissolves relatively quickly in the γ matrix, but substantial time (usually many hours) is required to obtain a uniform distribution of alloying elements via diffusion processes.

The objective of the present work is the metallurgical characterization of Waspaloy including its chemical analysis, and its characterization by optical microscopy, scanning electron microscopy and EDX analysis, in order to determine if the as cast ingot shows a heterogeneous microstructure and variations in chemical composition (segregation), grain size and hardness, from the external surface to the center of the Waspaloy ingot.

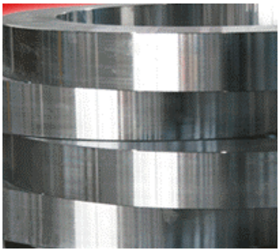


Figure 1. Waspaloy forged rings.

Experimental Procedures

Metallurgical characterization of the samples consisted of: chemical composition determination by ICP analysis, microstructure determination by optical and scanning electron microscopy and EDX, mean grain size measurement by image analysis, and Rockwell C hardness measurements. The material used in the present work was a 304.8 mm diameter hot-rolled bar of Waspaloy. It had a measured chemical composition showed in Table I.

Table I. Chemical composition of the Waspaloy ingot.

	C	**Mn**	**Si**	**S**	**P**	**Cr**	**Ni**
Nominal	0.03-0.10	0.0-0.10	0.0-0.15	0.0-0.015	0.0-0.015	18-21	Bal.
Measured	0.036	0.02	0.03	0.003	0.004	19.61	Bal.
	Co	**Fe**	**Mo**	**Ti**	**Al**	**B**	**Zr**
Nominal	12-15	0-2.0	3.5-5.0	2.75-3.25	1.2-1.6	.003-0.010	0.02-0.08
Measured	13.24	0.9	4.19	3.06	1.36	0.007	0.06

The microstructure of the as-received bar was examined in six representative locations, one at the outer diameter, 4 in the mid-radius, and one at the center. Figure 2 shows the sites where the samples were obtained from the Waspaloy ingot.

The samples were prepared for the purpose of obtaining optical micrographs to show the grain size, grain morphology, grain distribution, and second phases of the matrix. Figure 3 shows the micrographs obtained from the six samples, from the external diameter to the center.

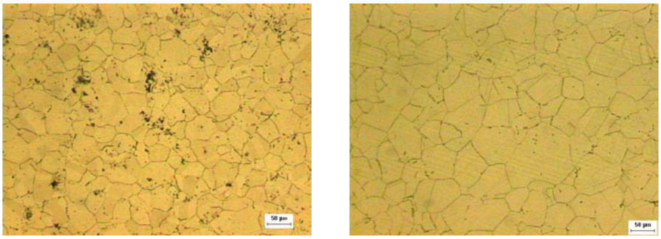
The grain size was measured by optical microscopy and image analysis, in accordance with ASTM Standard E-112. Table II and Figure 4 show the obtained results of grain size for samples M1 to M6.

Rockwell "C" hardness was measured on the external surface of the 6 samples. Table III and Figure 5 shows the hardness profile obtained from the outer surface (M1), to the center of the ingot (M6).

Figure 2. Location of the six samples obtained from the Waspaloy ingot.

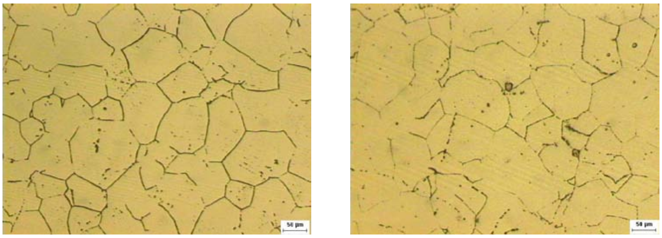
Table II. Average grain area and ASTM grain size values of samples

Sample #	1	2	3	**Average grain area (μm^2)**	**ASTM Grain size**
M1	1030.97	1647.51	1754.76	**1477.74667**	**6.5**
M2	1642.5	1681.36	2373.29	**1899.05**	**65.-6.0**
M3	2862	2084.38	3097.69	**2681.35**	**6.0-5.5**
M4	3302.5	3354.12	3456.6	**3371.07333**	**5.5-5.0**
M5	5323.6	5757.84	5326.56	**5469.33333**	**5.0-4.5**
M6	7000	7024.8	7839.93	**7288.24333**	**4.5-4.0**



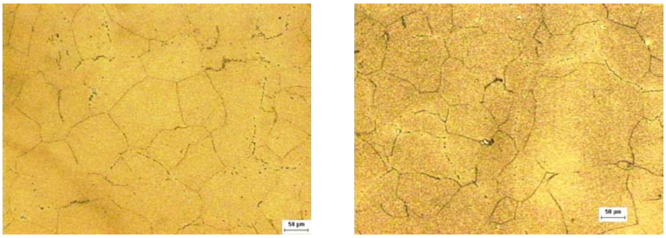
Sample 1 ingot outer surface

Sample 2



Sample 3

Sample 4



Sample 5

Sample 6 ingot center

Figure 3. Microstructures of the ingot, from the outer surface (Sample-1) to the ingot center (Sample-6).

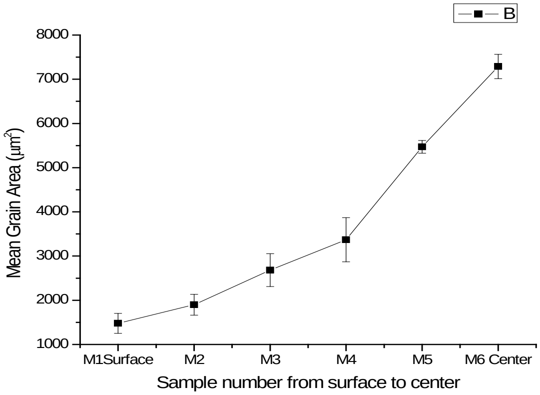


Figure 4. Average grain area and ASTM mean grain size.

Table III. Average Rockwell C hardness on the Waspaloy ingot samples

Sample	**M1**	**M2**	**M3**	**M4**	**M5**	**M6**
Rockwell “C” hardness	37	30.7	30	27.5	28	23
	36	32	29.8	26.5	26.2	24.5
	36	31.8	29	26.5	25	25
	35	32	29.4	25.5	25.8	25
	35	32	28.8	27.0	25.0	24
	35	31.5	28	25.3	25.0	25
	35	31	27	26	26.0	25
	34	31	27.3	25.5	24.8	25.5
	34	29	26	26	28.0	25
Mean value	**35.22**	**31.22**	**28.36**	**26.2**	**25.97**	**24.66**

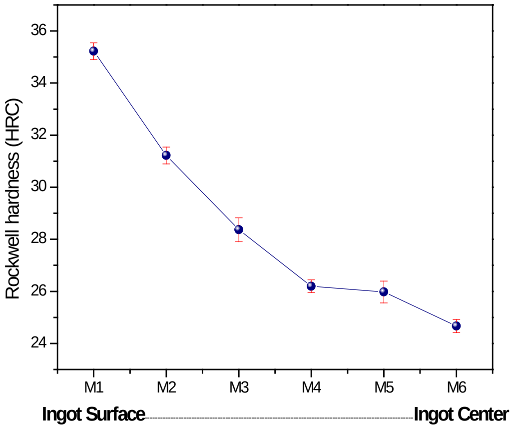
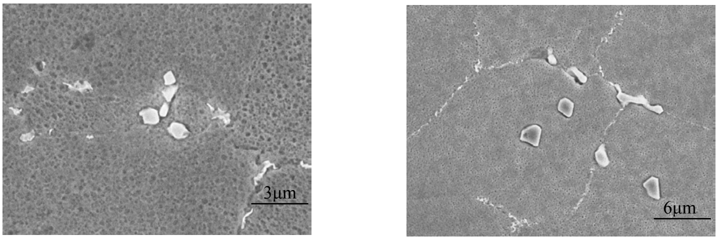


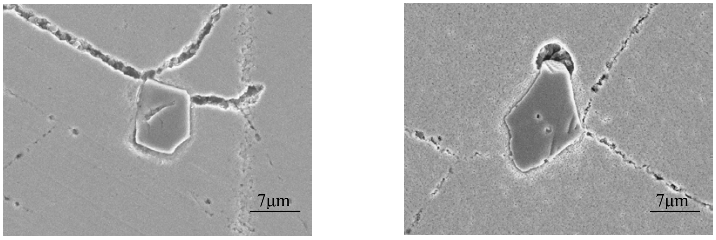
Figure 5. Average Rockwell C hardness measured on the Waspaloy ingot samples.

The microstructure was determined by scanning electron microscopy and EDX, identifying the phases that were present in the matrix, such as blocky carbides located inside the grains, and tiny carbides located on the grain boundaries. Figure 6 shows the microstructures of the six samples, from the outer surface to the center of the ingot.

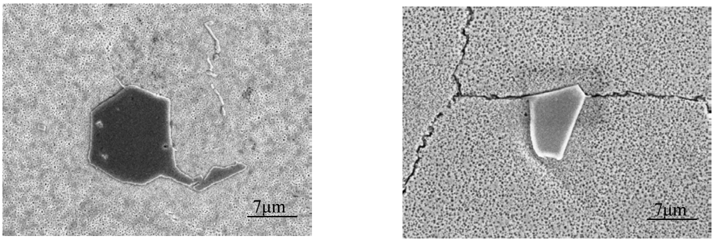
EDX analyses were performed in order to identify the carbides that are present in the microstructure of Waspaloy ingot. Grain boundary carbides contain carbon, molybdenum, titanium and chromium. Coarse carbides contain carbon, titanium and molybdenum, as shown in Table IV.



Sample M1 Sample M2



Sample M3 Sample M4



Sample M5 Sample M6

Figure 6. Scanning electron micrographs of the six samples obtained from the Waspaloy ingot, from outer surface (M1) to the ingot center (M6), showing coarse carbides of Mo and Ti, and carbides located on the grain boundaries.

Table IV. Elements detected in coarse carbides, grain boundary carbides and matrix

Sample	**C**	**Al**	**Mo**	**Ti**	**Cr**	**Fe**	**Co**	**Ni**
M1 Coarse carbides	**13.46**	2.27	6.21	3.35	**18.25**	1.15	11.10	44.21
M2 Coarse carbides	**23.50**	--	**21.04**	**51.17**	**1.62**	--	--	2.66
M3 Coarse carbides	**6.59**	--	--	**93.41**	**--**	--	--	--
M4 Coarse carbides	**19.17**	--	6.24	**74.60**	**--**	--	--	--
M5 Coarse carbides	**7.56**	--	--	**92.44**	**--**	--	--	--
M6 Coarse carbides	**19.33**	--	9.94	**60.79**	**2.62**	--	2.01	5.31
M1 grain boundary carbides	**14.82**	1.03	4.60	2.55	**19.91**	1.23	**11.94**	43.91
M2 grain boundary carbides	**24.85**	--	**17.04**	**36.63**	**5.96**	--	3.22	12.30
M3 grain boundary carbides	**26.69**	0.43	**18.75**	**37.23**	**4.27**	0.54	2.56	9.53
M4 grain boundary carbides	**23.82**	--	**18.90**	**45.26**	**3.20**	--	2.19	6.64
M5 grain boundary carbides	**17.63**	0.66	**14.24**	**24.52**	**10.58**	1.17	6.59	24.61
M6 grain boundary carbides	**22.26**	0.57	**16.96**	**35.78**	**5.62**	1.15	3.93	13.72
M1 matrix	**3.32**	1.27	4.40	3.01	**18.17**	1.35	**13.54**	**54.96**
M2 matrix	**3.34**	1.04	4.50	2.72	**18.61**	1.39	**13.53**	**54.89**
M3 matrix	**3.85**	1.30	4.40	3.05	**18.32**	1.27	**13.54**	**54.28**
M4 matrix	**3.93**	1.21	4.54	2.59	**18.35**	1.28	**13.76**	**54.35**
M5 matrix	**3.05**	1.34	4.42	2.76	**19.18**	1.46	**13.29**	**54.50**
M6 matrix	**9.52**	1.28	3.58	2.65	**17.14**	1.23	**12.99**	**51.61**

Results and Discussion

Chemical Composition

The chemical composition of the Waspaloy ingot meets the ranges specified in nominal composition, and it is within the range specified in Table I. There are no significant variations in the chemical composition among the six analyzed samples, as shown in Table IV.

Microstructure

The grain size on the external surface of the Waspaloy ingot is smaller than the grain size at the center of the ingot, as shown in Table II and Figures 3 and 4. This variation in grain size is due to different cooling rates on the external surface and the center of the Waspaloy ingot. The center of the ingot has a lower cooling rate than that of the external surface.

The microstructure of Waspaloy constitutes of a nickel matrix (γ), a γ′ phase as fine precipitates, and a minor amount of carbide grains which occur at the grain boundaries and in the nickel matrix [2]. Figure 6 shows the different carbide grains located on the grain boundaries and inside the nickel matrix; these carbide grains were analyzed by scanning electron microscopy and EDX to determine their compositions. Table 4 shows the detected elements in each type of carbide; the blocky carbides are composed principally of titanium (92-93 wt.%) and in some cases molybdenum (6-21 wt.%), titanium (51-74 wt.%) and chromium (1-18 wt.%). Carbides located at the grain boundaries contain titanium (24-45 wt.%), molybdenum (4-18 wt.%) and chromium (3-19 wt.%).

Hardness

The Rockwell "C" hardness of the sample located on the external surface (35 HRC) of the ingot is higher than the hardness of sample located at the center (24 HRC), as shown in Table III and Figure 5.

Variations of microstructure, such as grain size and the phases present, may cause a non-uniform strain distribution on forged rings, causing cracks in zones of different grain size, specially when the geometry of the ring is complicated with zones of different thicknesses.

Conclusions

- There is no significant variation in the chemical composition among the six samples of Waspaloy ingot analyzed.
- The microstructure shows variations in grain size from the external surface to the center of the ingot; the mean grain size on the external surface is smaller than that of the ingot center. The grain size distribution is not homogeneous within the same sample analyzed.
- Rockwell "C" hardness is lower in the center of the ingot than that of the external surface.

Acknowledgments

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