THE MICROSTRUCTURAL RESPONSE OF AS-HIP P/M U-720

TO THERMOMECHANICAL PROCESSING

J. M. Hyzak, R. P. Singh, J. E. Morra, T. E. Howson

Wyman-Gordon Company North Grafton, Massachusetts

## Abstract

Udimet 720 is a high strength nickel-base superalloy which in this case was produced via powder metallurgy processing; the billet was in the as-HIP condition. The microstructural response was studied as a function of thermal exposure in the range of  $1080^{\circ}$ C to  $1162^{\circ}$ C. Compression specimens were deformed in a matrix of five different temperatures ( $1050^{\circ}$ C -  $1140^{\circ}$ C) and five strain rates ( $0.006 \text{ min}^{-1}$  -  $3.0 \text{ min}^{-1}$ ). Resultant grain size,  $\gamma'$  precipitate volume fraction and propensity for cracking were evaluated. The grain size coarsened, as expected, with increased temperature for both the thermal exposure and deformed samples. Grain size also coarsened as the deformation strain rate was reduced. Grain boundary cracking (location and density) varied with deformation temperature and strain rate.

The resultant grain size correlated well with the volume percent primary  $\gamma'$  present in the microstructure. A relationship between the percent  $\gamma'$  in the structure, and the thermal exposure and deformation conditions is presented along with a discussion of grain boundary pinning. It is suggested that the primary  $\gamma'$  and the prior particle boundaries (ppbs) have significant influences on the resultant grain sizes.

## Introduction

Udimet 720 is a high strength nickel-base superalloy that is being utilized for turbine disk applications (1, 2). Forging stock can be cast-wrought billet or powder metallurgy product in either the as-HIP or extruded conditions. To properly design the forging and heat treat processes for these alloy forms, their thermomechanical response needs to be understood. This, of course, will vary not only with the billet form (cast-wrought, P/M) but also with prior processing (cogged, as-HIP, extruded). In this study, as-HIP P/M U-720 billet was used. The microstructural response to thermal exposure and to high temperature isothermal deformation has been examined.

### Material

U-720 powder was produced via an argon atomization process. -100 mesh powder was consolidated by HIPing at 1100°C and 103 MPa; 30.5 cm diameter billet was produced. The chemical composition of the powder blend is shown in Table I. The  $\gamma'$  solvus temperature of the billet determined by DTA was 1153°C. The as-HIP microstructure had a uniform grain size of ASTM 12 or finer with 10-20% of the structure unrecrystallized as large as ASTM 8. This structure is shown in Figure 1. The unrecrystallized grains are actually powder particles with an as-cast  $\gamma'$  structure. The powder particle boundaries (ppbs) are decorated with a fine network of particles believed to be predominantly carbides and oxides (3, 4).

Table I Chemical Co	mposition; Ma	i jor El	lements (	Wt. 9	8)
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С	0.02	Co	14.65	Ti	5.12
Si	0.02	Cr	17.74	W	1.33
Al	2.52	Fe	0.06	Zr	0.036
В	0.033	Mo	3.02		



Figure 1 - (a) Microstructure of as-HIP P/M U-720 billet, and (b) higher magnification of prior powder particle.

	Avg. G.S.	
Temperature*	(ASTM)	ALA**
As HIDed	>12	9
1080°C (1975°F)	>12	8
1100°C (2012°F)	>11	8
1120°C (2048°F)	11	8
1140°C (2084°F)	9 - 10	7
1149°C (2100°F)	7	5.5
1162°C (2125°F)	6.5	5.5
**ALA - As Large As	*2 hours a	t temperature

Table II Grain Size After Thermal Exposure

# Thermal Exposure

Samples with the as-HIP microstructure were exposed to various temperatures for 2 hours and air cooled. The objective was to characterize static recrystallization, grain coarsening and the development of thermal induced porosity (TIP). The results are presented in Table II. The average grain size coarsened beginning with exposure to  $1100^{\circ}$ C. At this temperature the grain size coarsened from ASTM 12 to ASTM 11, and at the highest temperature of  $1162^{\circ}$ C the grain size coarsened to ASTM 6.5. Representative photomicrographs are shown in Figure 2. Recrystallization of the remnant powder particles did not fully occur even at  $1120^{\circ}$ C. The maximum pore diameter increased from approximately 2  $\mu$ m in the as-HIP condition to 29  $\mu$ m at  $1162^{\circ}$ C.





Figure 2 - Grain structure of thermally exposed samples after two hours at a) 1140°C and b) 1162 °C.

	-	TEMPE	RATURE	S	
<u>Strain Rate</u>	<u>1050°C</u>	<u>1080°C</u>	<u>1100°C</u>	<u>1120°C</u>	<u>1140°C</u>
0.006 min <sup>-1</sup>	>12 ALA 8.0	11 8.0	9.5	8.0	1-2
0.025 min <sup>-1</sup>	>12 8.0	12 8.0	10 8.0	9	1-2
0.1 min <sup>-1</sup>	>12 8.0	>12 8.0	11 8.0	10 8.0	4.0
0.5 min <sup>1</sup>	>12 8.0	>12 8.0	12 8.0	10.5	5.0
3.0 min <sup>-1</sup>	>12 8.0	>12 8.0	12 8.0	11 9.0	7.0

Table III ASTM Grain Size of Compression Samples

### Deformation Studies

Compression tests were performed on small specimens measuring 10.1 mm in diameter and 15.2 mm high. The dies were maintained at the prescribed forge temperature; the specimen was brought to temperature and held for fifteen minutes prior to the initiation of testing. The specimens were upset 50% in height to a true strain of 0.7 and were then water quenched off the dies.

Twenty-five specimens were deformed in a matrix of five different temperatures ( $1050^{\circ}C$ ,  $1080^{\circ}C$ ,  $1100^{\circ}C$ ,  $1120^{\circ}C$ ,  $1140^{\circ}C$ ) and five different strain rates ( $0.006 \text{ min}^{-1}$ ,  $0.025 \text{ min}^{-1}$ ,  $0.1 \text{ min}^{-1}$ ,  $0.5 \text{ min}^{-1}$ ,  $3.0 \text{ min}^{-1}$ ). Specimens were sectioned in half and the grain size was rated at the center. The average as-forged grain sizes and as large as (ALA) grain sizes are listed in Table III. The grain size generally coarsened as the deformation temperature increased and the strain rate decreased. The effect of strain rate was small at the lower temperatures of  $1050^{\circ}C$  and  $1080^{\circ}C$ , but at the higher temperatures there was a large difference in the final grain size between the slowest and fastest strain rates. At  $1100^{\circ}C$  and  $1120^{\circ}C$ , there was a three grain size difference, and at  $1140^{\circ}C$  there was a range of five ASTM numbers (ASTM 2 - ASTM 7). It should be noted that the prior powder particles that made up the  $10 - 20^{\circ}$  of the as-HIP microstructure did not undergo recrystallization even when deformed at  $1100^{\circ}C$  to an upset of  $50^{\circ}$ .

There was also a significant effect of the deformation conditions on the grain boundary cracking in the as-forged upsets. First, the cracking varied across the compression upsets from the center to the surface (bulge) at mid thickness. The cracks in the bulge region were more prevalent and of greater size than in the center. In this bulge region, the cracks (number and size) at the lower deformation temperatures increased as the strain rate increased, and at the higher temperatures the cracking increased as the strain rate decreased. Photomicrographs are presented in Figure 3 for the extreme cases. It can be seen that the morphology of cracking

is different in the two cases. At 1050°C and the fast strain rate, the fracture occurred along prior particle boundaries, whereas, at 1140°C and the slow strain rate the cracks formed at the large grain boundaries developed during deformation.



Figure 3 - Grain boundary cracking after deformation at a) 1050°C and 3.0 min<sup>-1</sup>, and b) 1140°C and 0.006 min<sup>-1</sup>.

## Discussion

The thermal exposures and compression testing have demonstrated the large variation in microstructure that can be attained in as-HIP P/M U-720 while still in the subsolvus temperature regime. The as-HIP grain size was ASTM 12 with 10-20% of the structure made up of prior powder particles rated as ASTM 8. With a 50% upset at temperatures as high as 1100°C, these particles did not recrystallize.

There was a significant difference in coarsening behavior between the thermally exposed and deformed samples. The grain size of the as-HIP billet coarsened from ASTM 12 to ASTM 9 with subsolvus thermal exposures up to  $1140^{\circ}$ C. The grain size coarsened further to ASTM 6.5 ALA 5.5 with a supersolvus heat treatment at  $1162^{\circ}$ C for 2 hours. The grain size of the as-deformed samples, however, coarsened to ASTM 7 when forged subsolvus ( $1140^{\circ}$ C) at a strain rate of 3 per minute and to ASTM 1 - 2 when deformed at 0.006 per minute at the same temperature. The mechanism that controls the grain size in these situations is of interest especially in light of current design requirements where a uniform grain size of ASTM 8 - 10 may provide the best balance of low cycle fatigue and high temperature crack growth properties (2).

It is well documented that carbides at ppbs,  $\gamma'$  precipitates and oxides can effectively control the grain size in a structure by acting as pinning sites (5, 6). The ppb network, for example, is extensive. This could be seen readily after forging U-720 at the highest temperature (1140°C) where the grains coarsened beyond the ppbs. Figure 4 shows a newly formed equiaxed grain boundary among the deformed ppbs decorated with small carbides.



Figure 4 - Newly formed grain boundary (arrow) within field of highly deformed prior particle boundaries.

In this study, a consistent correlation was established between the existing grain size and the amount of primary  $\gamma'$  present in the structure. This correlation held for both the thermally exposed samples and the deformed compression specimens. In both cases, the grain size coarsened as the amount of  $\gamma'$  decreased with increased temperature. The resultant grain size has been plotted as a function of volume percent  $\gamma'$  in Figure 5. The data are also included in Table IV.

The data show that the grain size is related to the amount of primary  $\gamma'$  in the microstructure irrespective of the thermomechanical processing route; the deformed specimens and those only thermally exposed fall in the same population. This suggests that  $\gamma'$  controls the grain size below the  $\gamma'$  solvus. Above the  $\gamma'$  solvus, however, the  $\gamma'$  is no longer present. In this case, it is thought that the ppbs control the grain size (ASTM 6 - 8) for the non-deformed samples. It has been frequently observed

		Compression Specimens Temperature (°C)				
Strain Rat	ate	1080	1100	1120	1140	
0.006/mir	n	29	16	4	0	
0.1/min				9		
3.0/min			28	16	6	
	The	ermal Exp Tempei	oosure S rature (	pecimens C°)		
1080	1100	1120	1140	1149	1163	
38	33	19	7	2	0	

Table IV Variation in Volume % Primary Y'



Figure 5 - Grain size as a function of volume percent  $\gamma'$  for both thermal exposure and deformed samples.

in our laboratory that P/M superalloys will coarsen to an ASTM 6 - 10 grain size when supersolvus heat treated and that this grain size is stable as a function of time at temperature. Those specimens deformed near the  $\gamma'$  solvus temperature, with no appreciable  $\gamma'$  present, however, behaved differently. They apparently had enough energy to break through these ppb pinning sites and established a much coarser grain size (ASTM 1 - 2).



Figure 6 - Volume percent  $\gamma'$  versus elevated temperature exposure.

Examining the data in Table IV reveals that the volume percent  $\gamma'$  present in the microstructure is dependent on the exposure temperature and the deformation strain rate, if forged. For a given temperature, the deformed specimens had less primary  $\gamma'$  than did the specimens given only a thermal exposure. In addition, the specimens deformed at the slow strain rate had less  $\gamma'$  than the specimens deformed at the same temperature but at a faster strain rate. The data are graphically presented in Figure 6. Representative photomicrographs of the  $\gamma'$  distributions are shown in Figure 7. It is realized that the comparison is somewhat confounded since the specimens deformed at slower strain rates were exposed to high temperatures for longer times. However, the specimens with only the thermal exposures were at temperature for 2 hours, longer than most of the deformed samples.

This observation of deformation enhanced dissolution may be tied to strain induced microstructural instabilities which result in enhanced diffusion. Menzies observed enhanced diffusion at slow strain rates in deformation of P/M IN-100 (7). In addition, the grain boundary mobility can also play an important role in determining the volume fraction and size of the  $\gamma'$  particles. A moving grain boundary can dissolve both  $\gamma'$  and carbide particles, hence reducing the volume fraction of these phases (8, 9, 10).

A dependence of grain boundary cracking on forging conditions has been observed. The larger effect in the bulge area of the upset can be understood in terms of the stresses generated during forging. The bulge section develops a large tensile stress while the center of the upset is under compression during the deformation. At low temperature and fast strain rates cracking occurred along prior particle boundaries (Figure 3). It appears that the powder particles do not deform and the boundaries are unable to accommodate the strain. This was observed also during isothermal forging of MAR M200 P/M compacts (11). At the highest temperature and slow strain rates, the newly developed coarse grain size was susceptible to grain boundary cracking. There was appreciably no primary  $\gamma'$  present in this structure to strengthen the grain boundary during sliding which is prevalent under these deformation conditions.

### Summary

This study has shown the variation in microstructure that can be generated in as-HIP P/M U-720 by altering the thermomechanical processing. The grain size and grain boundary cracking are both significantly affected by temperature and strain rate. The grain size coarsened to ASTM 9-10 after a thermal exposure at 1140°C and to a grain size of ASTM 6.5 at 1162°C for 2 hours. The deformed samples had a grain size of ASTM 7 when strained at 1140°C at a strain rate of 3.0 min<sup>-1</sup> and ASTM 1 - 2 when deformed at 0.006 min<sup>-1</sup> at the same temperature.

It has been shown that the percent primary  $\gamma'$  in the microstructure is also a function of temperature and strain rate, and the resultant grain size correlates well with the amount of  $\gamma'$  present for both the thermally exposed and deformed samples. However, the dissolution of  $\gamma'$  is strongly influenced by deformation, and the amount of primary  $\gamma'$  decreases as the deformation temperature is increased and the strain rate decreased. As the amount of  $\gamma'$  approaches zero the grain size is pinned at the ppb size (ASTM 6 - 8) for the thermal exposure but grows to ASTM 1 - 2 when deformed.

Grain boundary cracking was also noted as a function of temperature and strain rate. Cracking occurred at ppbs at low temperatures and high strain rates and at large grain boundaries at high temperatures and slow strain rates.



Figure 7 - Primary Y' distribution for samples: a) 1100°C exposure b) 1140°C exposure c) 1100°C, 3.0 min<sup>-1</sup> d) 1140°C, 3.0 min<sup>-1</sup> e) 1100°C, 0.006 min<sup>-1</sup> f) 1140°C, 0.006 min<sup>-1</sup>