T E C H N I C A L U P D A T E

Stress Rupture Properties of Nickel-Base Superalloys with Equiaxed Grain Structure

Introduction

Without the proper controls, high-temperature properties of common nickel-base superalloys in the equiaxed condition can be extremely variable.

For example, in production qualification of IN 100 cast test bars, the 1800° F (980° C) and 29 ksi (200 MPa) stress rupture life varied from 28 to 64 hours with elongation varying from 3 to 14%. In actual part testing, the 1400° F (760° C) and 85 ksi (585MPa) stress rupture life of alloy IN 100 varied from 14 to 180 hours with elongation varying from 1.1 to 12%. Its 1800° F (980° C) and 29ksi (200 MPa) stress rupture life varied from 12 to 64 hours with elongation varying from 3 to 14%, even when the test bars and castings were made with good foundry parameter control.

Two of the large engine manufacturers have found similar variations when testing IN 713 and B 1900 castings. This update presents some information from Hitchiner files on 21 special melts of nickel-base alloys. These melts were made to investigate these variations and some production-generated information as well. Since these variations have been found on many different superalloys, it is expected that the data presented here for two alloys can be extended to other, similar alloys.

Alloy IN 100

Fourteen small vacuum heats were made to evaluate several factors—alloying practice, carbon content, raw materials, foundry practice and heat treatment.

Alloying Practice. To assure the charge stock was not a variable, we prepared charges for three melts by splitting one lot of materials into three charges. We then varied the three melts by maximum boiling, some boiling and no boiling at all before adding the more reactive elements (titanium, zirconium and aluminum). In testing, the 1800° F (980° C) and 29 ksi (200 MPa) stress rupture life varied from 23 to 29 hours. This result did not indicate a big effect on stress rupture life properties. However, if harmful volatile elements had been present in the charge materials, some effect may have been noted. For the balance of the melts, the intermediate boiling procedure was used.

Carbon Content. Again splitting the raw materials, we made five heats. Only the carbon content was varied, from 0.10 to 0.27%. There was no clear stress rupture life trend with varied carbon. The 0.10 % C melt stress rupture life was, however, short in the as-cast condition; heat treating, to be discussed, lengthened it up into the range of the higher carbon composition for the 1800° F (980° C) stress rupture life. Clearly, carbon variations are not the significant factor for the property variations.

Raw Materials. In an Edisonian fashion, we substituted molybdenum wire and graphite for the molybdenum rhondelles and high-carbon chrome normally used and cast all test bars from remelted pigs of the original melt. The result: The average 1800° F (980° C) stress rupture life extended from 29.5 hours to 40.

Since that looked promising, we substituted titanium scrap for the sponge normally used and pure vanadium for the aluminum vanadium in successive heats. Both improved stress rupture life to 44 hours. The average stress rupture life extended to 48.8 hours with all four substitutions. When we measured the 1400° F (760° C) and 85 ksi (585 MPa) stress rupture life, the improvement was even more dramatic—the new materials gave 245.5 to 422.1 hours life as compared to the regular materials range of only 59.5 to 119.3 hours.

Figures 1 and 2 are 1000X micrographs of the bars with the longest and shortest stress rupture lives. They do not show major differences between the two.

Oxygen and nitrogen also did not correlate with the improved stress rupture life properties. Oxygen varied from 4 to 32 ppm and nitrogen from 16 to 38 ppm. Grain size on the surface of the test bars varied from 1/32 inch (0.75mm) to 3/32 inch (2 mm), but did not correlate with property variation. Specified elements were in the required range and variation in controlled elements did not show correlation with the differences in stress rupture life. Finally, spectrographic analysis of the “better” raw materials did not show any significant differences. It is possible that the presence or absence of small amounts of some undetected element was the cause of the improved properties.

Foundry Conditions. An experienced foundry metallurgist directed all the melts, and we decided to vary some factors intentionally to assure that minor experimental variations had not caused the big variation in stress rupture life properties. We therefore cast some bars directly from the raw materials melt and some from remelted pigs of the first melt.

Comparing regular materials cast both ways showed an average of 26 hours stress rupture life for the 1800° F (980° C) for single melting and 32 hours for the double melting, possibly a small improvement.

To see if remelt time had an effect, we melted one heat of the “better” raw materials for 51 minutes

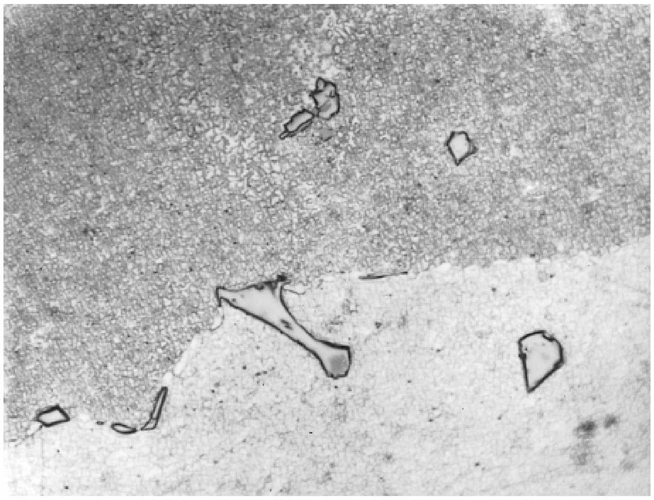

Figure 1. Cast structure of the test bar with the best stress rupture life properties. Etched with Marbles reagent, 1000X.

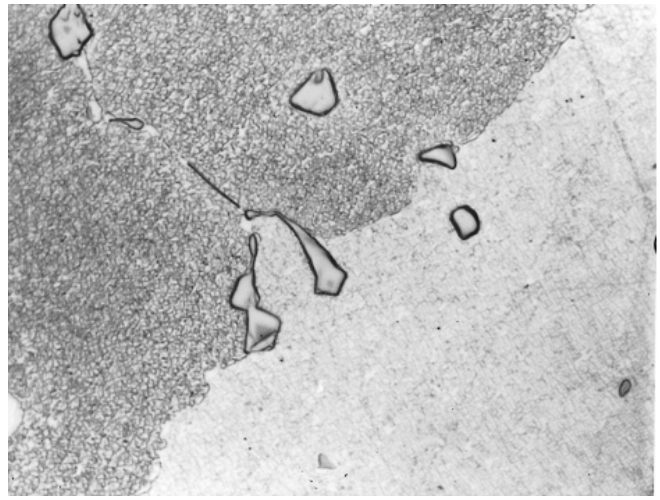

Figure 2. Cast structure of a test bar with low stress rupture life properties. Etched with Marbles reagent, 1000X.

instead of the usual 20. Both heats gave 1800° F (980° C) stress rupture life values ranging from 47.6 to 52.2 hours. One mold of bars was given a 1900° F (1038° C) preheat with 11 minutes from the preheat to cast as compared to the normal 1700° F (927° C) preheat with 20 minutes from oven to cast. Even with the new raw materials, the 1800° F (980° C) stress rupture life was only 37.5 hours average and the 1400° F (760° C) and 85 ksi (585MPa) stress rupture life averaged only 111.1 hours, essentially canceling the effect of the "better" raw materials.

Metallographic examination of these bars showed more segregation and porosity and a coarser internal grain structure even though the surface grain was smaller than some standard procedure bars. Raising the casting temperature from 2825° F (1552° C) and 2925° F (1607° C) had no significant effect. However, quick shakeout (5 minutes instead of 2 hours) caused the 1400° F (760° C) stress rupture life to drop dramatically when we heat treated the bars as described below. Thus, it was quite easy to destroy the effect of the "better" raw materials.

Heat Treatment. The common heat treatment for IN 100 was to thermal cycle it twice to 2000° F (1093° C) and then hold at 1200° F (649° C) and make the test bar data represent what was being put into the engines. While it had a major effect on fast-cooled bars, its

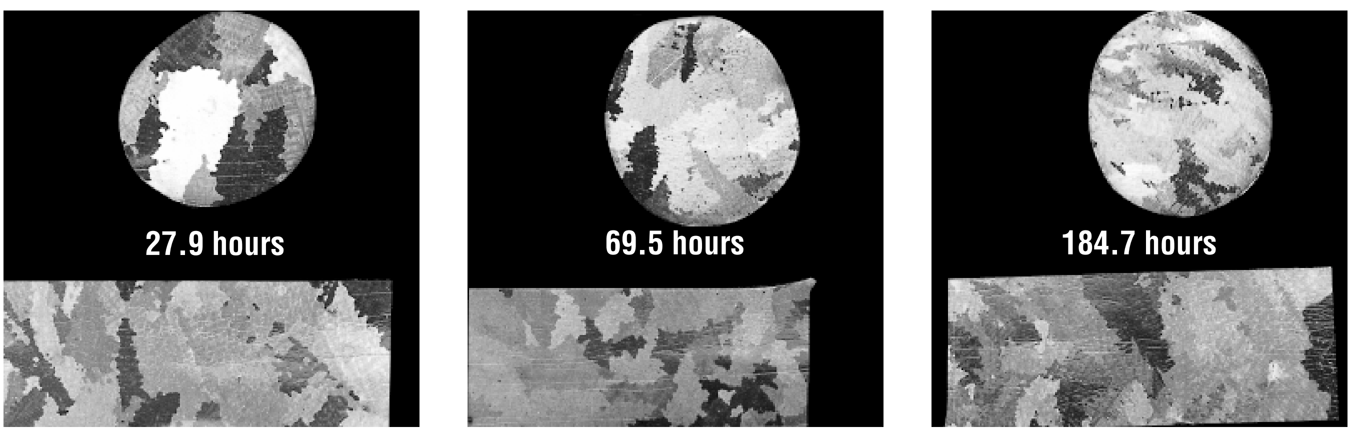

Figure 3. Internal grain variations and resulting 1400 °F (760 °C) and 85 ksi (585 MPa) stress rupture lives. Consider only transverse section as the longitudinal has random orientation to cast surfaces of the blades. (4X)

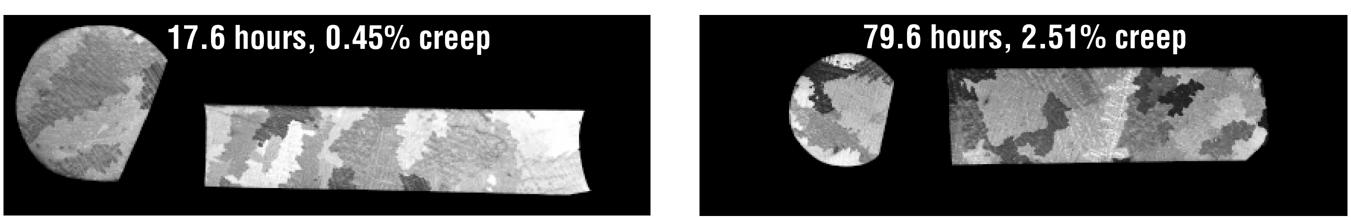

Figure 4. Two bars machined from the same blade side-by-side to eliminate many variables. Note the grain structure at the fracture area. The bar with the finer grain at fracture is clearly better. (2.6X)

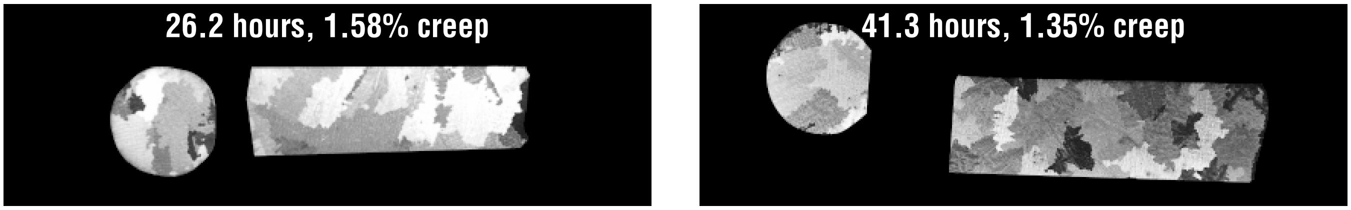

Figure 5. Bars from additional castings from the same mold used in Figure 4. Note the bar with the most grains in the fracture area has the best stress rupture life properties. (2.6X)

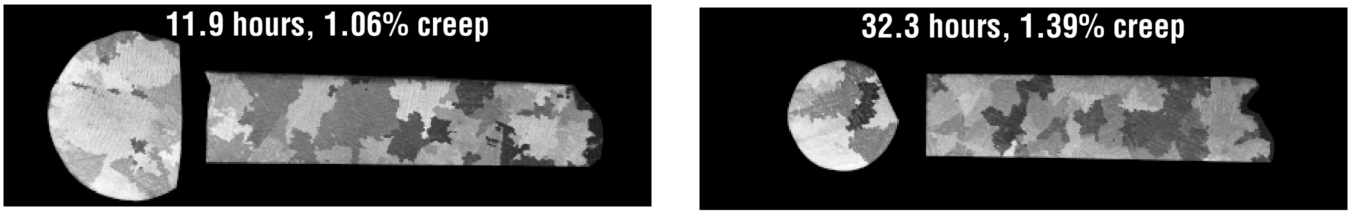

Figure 6. Bars from additional castings from the same mold used in Figure 4. Again, the bar with the most grains in the fracture area has the best properties. These bars were tested at 1400 °F (760 °C) and 90 ksi (619 MPa). (2.6X)

effect on all other test conditions was minor, possibly slightly improving the 1800° F (980° C) stress rupture life.

Since it appeared as if the raw materials had an important effect on properties, we made cast blades using the raw materials that gave good stress rupture life properties in test bars. Bars machined from castings did not show the improvement that the cast-to-size bars did when the cast-to-size bars were cast with the optimum foundry parameters. This suggested a more thorough investigation of the effect of grain structure itself.

Alloy B 1900

Internal Grain Size. In machined-from-blade (MFB) test bars, this alloy also showed a wide variation in stress rupture life properties in production testing. Its 1800 °F (980 °C) and 27 ksi (186 MPa) stress rupture life was 10 to 80 hours with 3 to 10% elongation; its 1400 °F (760 °C) and 85 ksi (585 MPa) stress rupture life was 10 to 200 hours with 0.25 to 5.2% elongation.

Since work with the IN 100 showed that an apparent effect of raw materials could be erased by changing casting parameters and the B 1900 showed a wide variation in properties due to the internal (not surface) grain size (Figure 3), we studied the effect of grain size in more detail.

Detailed Study. We machined a number of test bars from one mold of blades to separate many variables from the effect of grain size. Stress rupture is at 1400 °F (760 °C) and 85 ksi (585MPa) unless otherwise noted.

Figure 4 shows a huge variation in results between two bars machined from the same blade, side by side. Figures 5 and 6 show the results of four more bars from four additional castings from the same mold. Clearly, test bars with the most grains in their cross sections *at the fracture* gave the best properties. Other bars tested from the same mold yielded a total range of stress rupture life at 1400 °F (760 °C) of from four to 79 hours, essentially as much as found in production testing of many heats of blades.

Varied Grain Size Study. To further illustrate the pronounced effect of grain size, a mold of cast-to-size test bars was cast with a slow pour to create bars of varying grain size. This created some bars with very fine grain and some fairly normal, as can be seen in Figure 7. The very fine grained bars averaged only 6.3 hours life at 1400 °F (760 °C) and 90 ksi (619 MPa), while the normal grain size bars averaged 66.7 hours, clearly a grain size effect.

Most test bars were machined from the center of the cast bars. The grain is considerably different away from the center, even in the normal bar, as can be seen in Figure 7. Subsequently, we compared three bars from one mold with grain size like the bottom bar of Figure 7 at 1400 °F (760 °C) and 94 ksi (647 MPa) by machining one from the center (normal procedure) and two away from the center. The center bar had an 87.4-hour life. The two bars machined away from the center had lives of zero and 1.4 hours. These results again showed the huge effect of grain structure.

All of this work led us to be able to control the grain size to achieve good properties using a certain

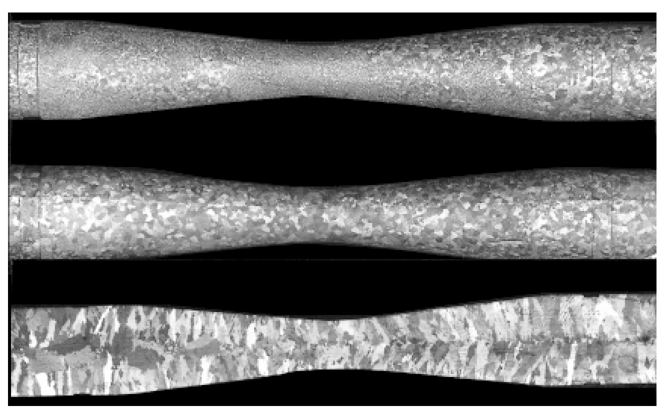

Figure 7. Two types of grain produced from a mold cast with a slow pouring speed. The bars with the really fine grain in the gage length are illustrated by the top bar. (1X)

procedure for testing a given blade in B 1900, Figure 8. Controlling to a finer grain size in the test bar area eliminated the low values in the test bar area. This probably gave more of an [001] direction parallel to the test direction as well. Better properties in such a local area do not assure all areas of the blade are of better quality, however.

Fundamental Cause of Variation of High-Temperature Properties

1. Much work has been done evaluating the high-temperature properties of single crystal nickel alloys and the effect of the crystallographic direction of testing on such properties. For example, for alloy MAR-M-200, at 1400 °F (760 °C) and 100 ksi (689 MPa) the stress rupture life will vary from 5.0 hours when tested in the [011] direction to 1914 hours when tested in the [001] direction. Testing at 1800 °F (980 °C) and 30 ksi (207 MPa) gives a variation of 79.4 to 147.6 hours with orientation, a large variation but less than at 1400 °F (760 °C). Thus, the load a particular crystal can carry depends on its orientation to the stress direction, especially at 1400 °F (760 °C).

2. As can be seen from the figures, especially the lower two bars of Figure 7, "equiaxed" castings consist of fine surface grains, which go to coarser columnar grains growing toward the thermal center. Often there are equiaxed grains in the centerline. Such structures can be controlled in a casting of a given shape. However, depending on the size of the grain and the size of the test bar, it may be possible to get only a few grains in a cross section. If one or two of these grains have a crystallographic direction unfavorable to the stress direction, they will not support the loads, which shifts the load to more favorably oriented grains, if there are any, overloading them and causing short lives. Since the weaker grains won't support the load, triaxial stresses are generated between the grains, causing lower elongations.

If all grains happen to be favorably oriented, high stress rupture life properties result. If the grains are fine and truly equiaxed through the failure area (top bar, Figure 7), there will always be enough unfavorably oriented grains to generate low stress rupture life properties.

Thus, cutting a small bar out of a casting consisting of thousands (or millions) of grains of various sizes and orientations to the test direction bears little or no relationship to the capability of that particular casting as a whole part. Test data generated from such bars is highly sensitive to process variation in making a given casting, which does, however, make the testing a valuable measure of process control.

Use of equiaxed castings. Equiaxed castings are inherently lower in high-temperature properties than the best directional properties of columnar or single crystal castings, but they are much lower in cost and can be made to tight metallurgical control. Since they consist of so many crystals, the castings can give uniform performance. However, the extreme mechanical property anisotropy of the nickel-base alloy crystal requires that the grain size be controlled for any part that is used to the full capabilities of these remarkable alloys.

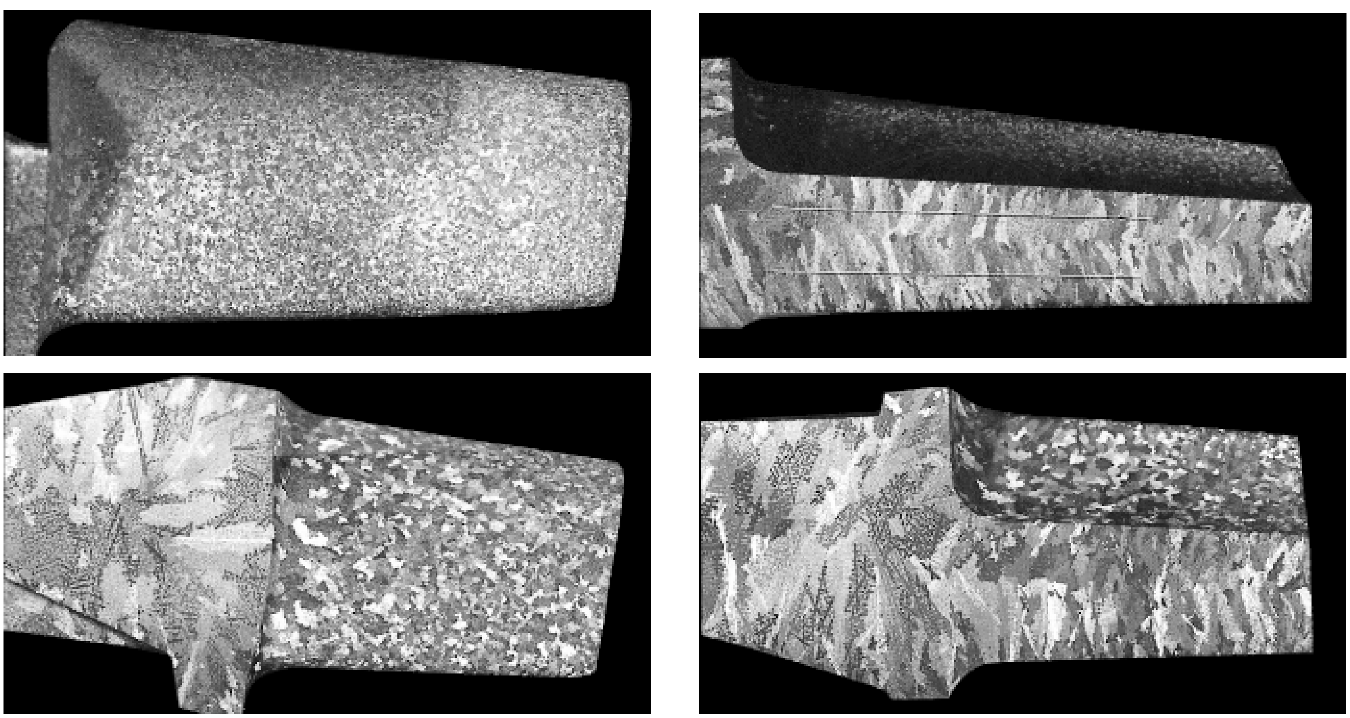

Figure 8. Top: Surface and internal grain that yielded the longest stress rupture lives—80 to 120 hours—at 1400 °F (760 °C) and 85 ksi (585 MPa). Bottom: The more equiaxed and coarser internal grain structure yielded shorter stress rupture lives—just 40 to 100 hours. (2X)