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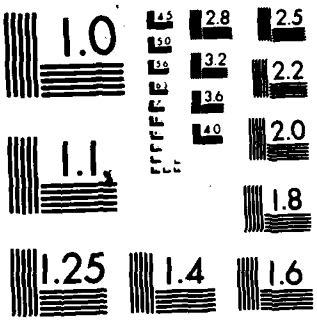
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Initial efforts were addressed at the preparation of powders that would result in the development of HIP'd logs possessing a large range of grain sizes and grain size distributions. After several trials, it was determined that the widest range of alloy grain size could be obtained by using different HIPing temperatures in conjunction with different solution treatments (ST). The following treatments and grain sizes were identified: [1107°C HIP + 1121°C ST (ASTM #13)]; [1107°C HIP + 1149°C ST (Duplex size ASTM average 8-9)]; [1107°C HIP + 1177°C ST (ASTM #6-6.5)]; [1246°C HIP + 1149°C ST (ASTM #4)]. Further aging treatments are planned at 650°C and 800°C to obtain fine and coarse γ' precipitate sizes, respectively.

The decision was made to use the disc-compact (D)CT specimen for fatigue crack propagation experiments; this configuration is convenient for use with the cylindrically-shaped HIP'd logs and results in considerable savings in machining costs. The existing computer-controlled test program was then modified to include the compliance coefficients for the (D)CT specimen. Preliminary experiments have verified the accuracy of this program modification. The model

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developed to account for grain size effects during cyclic overloads at low ΔK levels was tested successfully with data reported in the open literature. It was found that cyclic delay at low ΔK levels, resulting from a given overload cycle, increased with increasing grain size and this effect was rationalized in terms of changes in the effective overload ratio.

Initial fatigue crack propagation tests were completed with both material and computer-controlled test system performing in a well behaved manner. Crack closure measurements were obtained which allowed crack growth rate data to be compared with ΔK_{eff}. Grips and fixtures have been designed and ordered which will permit cyclic strain experiments to be conducted at both room and elevated temperatures. Techniques were developed to prepare thin foils to allow for detailed examination of alloy microstructures and cyclic load-induced dislocation structures.

PROJECT TITLE: "A Study of Fatigue Crack Propagation in Powder Metallurgy Hot Isotatically Pressed Nickel-Base Alloy"

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Powder Preparation

Initial efforts were addressed at the preparation of powders that would result in the development of HIP'd logs possessing a large range of grain sizes and grain size distributions. LC Astroloy powder was fractionated to -100/+120, -140/+170, -230/+325 and -325 mesh, and heat treated in H_2 at 1096°C (2005°F)/1 hr. H_2 cool, 1166°C (2130°F)/1 hr./ H_2 cool, and 1166°C (2130°F)/4 hr./H_2 cool. The developed grain sizes did not appear to be a function of particle size. A grain size range of 10-30 μm resulted from the lowest temperature heat treatment. Grains coarsened to 70-100 μm at 1166°C (2130°F)/1 hr., while a higher incidence of twinning and no appreciable grain growth occurred with the 1166°C (2130°F)/4 hr. hold time.

As a second test, the fractions between -100 and +325 mesh were combined as a 50:50 blend of the 1096°C (2005°F) and 1166°C (2130°F)/ 1 hr. heat treatments. The powder was then HIP'd at 1093°C (2000°F). A combination of the original fine and coarse grains resulted. Some coarsening of the microstructure occurred with additional heat treating at 1121°C (2050°F)/1 hr.

In comparison, a sample of -140 mesh Astroloy, previously HIP'd at 1160°C (2120°F) was examined and found to have a microstructure very similar to that of the HIP mixture described above. An additional heat treatment of 1 hr. at 1166°C (2130°F) produced total recrystallization but no further grain growth.

These results suggest that size fractionation does not provide any control over final grain size. Similarly, close grain size control is not obtainable for a given heat treatment; a variety of grain sizes results. It has been decided, therefore, that samples for this study will be based on -140 mesh powders that are to be HIP'd in a range between 1107°C (2025°F) and 1246°C (2275°F) and then solution treated at different temperatures to achieve different grain sizes. From discussions with CarTech personnel and Dr. Robert VanStone, from the General Electric Co., it is believed that the 1246°C (2275°F) HIP treatment (i.e., above the γ' solvus temperature) will allow for considerable grain growth while at the same time inhibiting the formation of thermally induced porosity (TIP).

Samples HIP'd at 1107°C (2025°F) (-140 mesh powders) were then given additional solution treatments to explore the range of grain sizes that could be obtained with this HIPing condition. As HIP'd, the material reveals a grain size range of ASTM 13-14 associated with the presence of coarse non-strengthening γ' phase. When the material was subsequently solution treated for 2 hrs. at 1121°C (2050°F), the grain size did not change to any significant degree though the γ' phase was present in a much finer size and distribution. A duplex grain size distribution with an average grain size of ASTM 8-9 was achieved when the as-HIP'd material was solution treated at 1149°C (2100°F) for 2 hrs. Since this latter heat treatment is above the γ' solvus temperature, no γ' phase was observed when the material was viewed in the optical microscope. Significant grain growth occurred when the as-HIP'd material was heat treated at 1177°C (2150°F)

Fatigue Test Results

Two fatigue crack propagation (FCP) tests were conducted using the disc-shaped sample. Both specimens were HIP'd and solution treated at 1149°C (2100°F); one sample was aged at 800°C for 24 hrs. to achieve a coarse γ' distribution and the second sample was aged at 650°C for 24 hrs. to achieve a fine γ' distribution. Figure 2 shows the results of the coarse γ' sample which was tested at a load ratio (R) of 0.1 under computer-controlled K-increasing and K-decreasing conditions. Figure 3 reveals the results for the fine γ' material that was tested at R=0.5. Since both γ' and R levels were different in the two tests, no direct comparison of results can be made at this time.

During the course of these tests, crack closure (K_{op}) measurements were obtained to allow crack growth rates to be compared as a function of K_{eff} ($K_{max}-K_{op}$). The two sets of data in Figure 3 correspond to ΔK_{app} (□) and ΔK_{eff}(+) values, respectively. Note that much of the break in the curve of ΔK_{app}-da/dN is eliminated when the data are plotted in terms of ΔK_{eff}.

Room Temperature Behavior - Cyclic Strain

The influences of the microstructural changes on the deformation are most clearly examined in specimens in which the deformation history is defined. For cyclic loading conditions this is most easily achieved using low cycle fatigue experiments in which the imposed plastic strain is controlled. Tests of this nature will permit the determination of the cyclic mechanical properties and by the examination of specimens prior to failure the observation of the microstructural influences on the deformation structure. Thin foils will be used for the latter purpose with specific attention focussed on the effects of changes in the precipitate distribution and the resulting dislocation structure. The interaction of dislocation structures with second phase particles and grain boundaries will also be made. Observations of this type will enable a more critical appraisal of the role of the microstructural features in the crack propagation characteristics to be made.

To facilitate these observations it has been necessary to design and manufacture accessories for the present fatigue machines. Loading bars capable of self-alignment under alternating loads have been designed and assembled together with extensometry which will enable the determination and control of the applied cyclic strain.

Elevated Temperature Fatigue Behavior

As has been previously stated, the influence of the dispersed phases and the grain boundaries is likely to be different at elevated temperature. Under these circumstances an examination of the low cycle fatigue deformation structure would once again be beneficial. The work would be extended to encompass the influence of the waveshape on the deformation behavior.

In order that these examinations of the material's behavior may be made, a furnace that can be attached to the present fatigue machine for 2 hrs. This treatment resulted in an average ASTM grain size of 6-6.5. Longer heat treatment times (i.e., 24 hrs.) at this temperature did not result in additional grain growth; rather, a large number of annealing twins were noted. Samples HIP'd at 1246°C (2275°F)* and solution treated at 1149°C (2100°F) for 2 hrs., developed a relatively coarse, average grain size of ASTM 4 with no apparent TIP structure.

In summary, several heat treatments and HIPing conditions have been identified that will produce a wide range of grain sizes. These treatments are summarized in Table I.

Some preliminary aging studies have been attempted. Samples were aged at 800°C (1472°F) for 24 hrs. and also at 650°C (1202°F) for 5 hrs. The resulting γ' sizes were found to be coarse and fine, respectively, as expected.

Test Procedures

In planning for the long crack-fatigue crack propagation (FCP) phase of this program, efforts were made to modify the automation package for the computer system which is an Instron Corporation supplied Digital PDP 11/03 computer with the RSX-11M operating system. The Instron supplied FCP program supports both CT and WOL geometries but not the disk compact sample (D)CT. It was necessary, therefore, to modify the program to include compliance coefficients for the (D)CT specimen. These coefficients were determined by Robert Vecchio during this reporting period (see Appendix A), based on the theoretical and experimental results of Newman and Fisher. These results are to be submitted for publication in the near future.

Literature Data Analysis

Based on the results of Vecchio and this writer,[1,2] regarding the influence of grain size and yield strength of delay cycles after an overload, an attempt was made to normalize the delay results published recently by Antolovich and Jayaraman.[3] They noted that the amount of cyclic delay following a 50% overload decreased with increasing ΔK level (plane strain controlled behavior) and varied with grain size and γ' particle size. The data from this paper were analyzed with the cyclic yield strength being approximated by the flow stress

$$(\sigma_{flow} = \frac{\sigma_{TS} + \sigma_{ys}}{2}).$$

The overload plastic zone was then computed and normalized by the grain size with the results being shown in Figure 1. The normalization of these data is most encouraging in that it supports our fatigue model as it relates to the combined influence of microstructure and overload cycles on the fatigue life of engineering alloys.

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*Compacted at IMT Corporation in Andover, MA, through the courtesy of Dr. J. Runkle.

loading frames has been designed and ordered. The furnace is capable of operating at temperatures up to 1000°C for both crack growth specimens and low cycle fatigue specimens. In addition, a loading system capable of withstanding the elevated temperatures has been designed and ordered.

Educational Seminars

During the first four months of this contract, arrangements were made to invite several speakers to the Lehigh campus to lecture on topics of major importance to the grant objectives. In addition, the lecturers spent a considerable amount of time with the writer, graduate students and key personnel from CarTech Corporation who are preparing the Astroloy powders for this study. These discussions were most helpful in that they exposed us to recent developments in fatigue research. Dr. Robert VanStone, G.E. Corp., Evendale, Ohio spoke on fatigue fracture of gas turbine nickel-based alloys; Dr. Richard Gangloff, Exxon Corp., Linden, NJ, spoke on short crack fatigue fracture in nickel-based alloys; and Dr. George Yoder, NRL, Washington, D.C. spoke on metallurgical aspects of fatigue crack propagation in engineering alloys.

References

1. R. S. Vecchio, R. W. Hertzberg and R. Jaccard, Scripta Met., 17, 1983, 343

2. R. S. Vecchio, Master's Thesis, Lehigh University, 1983.

3. S. D. Antolovich and N. Jayaraman, Fatigue, Environment and Temperature Effects, J. J. Burke and V. Weiss, Eds., Plenum Press, New York, 1983, 119.

TABLE I

Proposed HIPing and Heat Treatments for This Research Work

HIP Treatment	Heat Treatment	Grain Size
1107°C/2 hrs. (2025°F)	1121°C/2 hrs. (2050°F)	ASTM 13
1107°C/2 hrs. (2025°F)	1149°C/2 hrs. (2100°F)	Duplex structure ›verall average ASTM 8-9
1107°C/2 hrs. (2025°F)	1177°C/2 hrs. (2150°F)	ASTM 6-6.5
1246°C/2 hrs. (2275°F)	1149°C/2 hrs. (2100°F)	ASTM 4

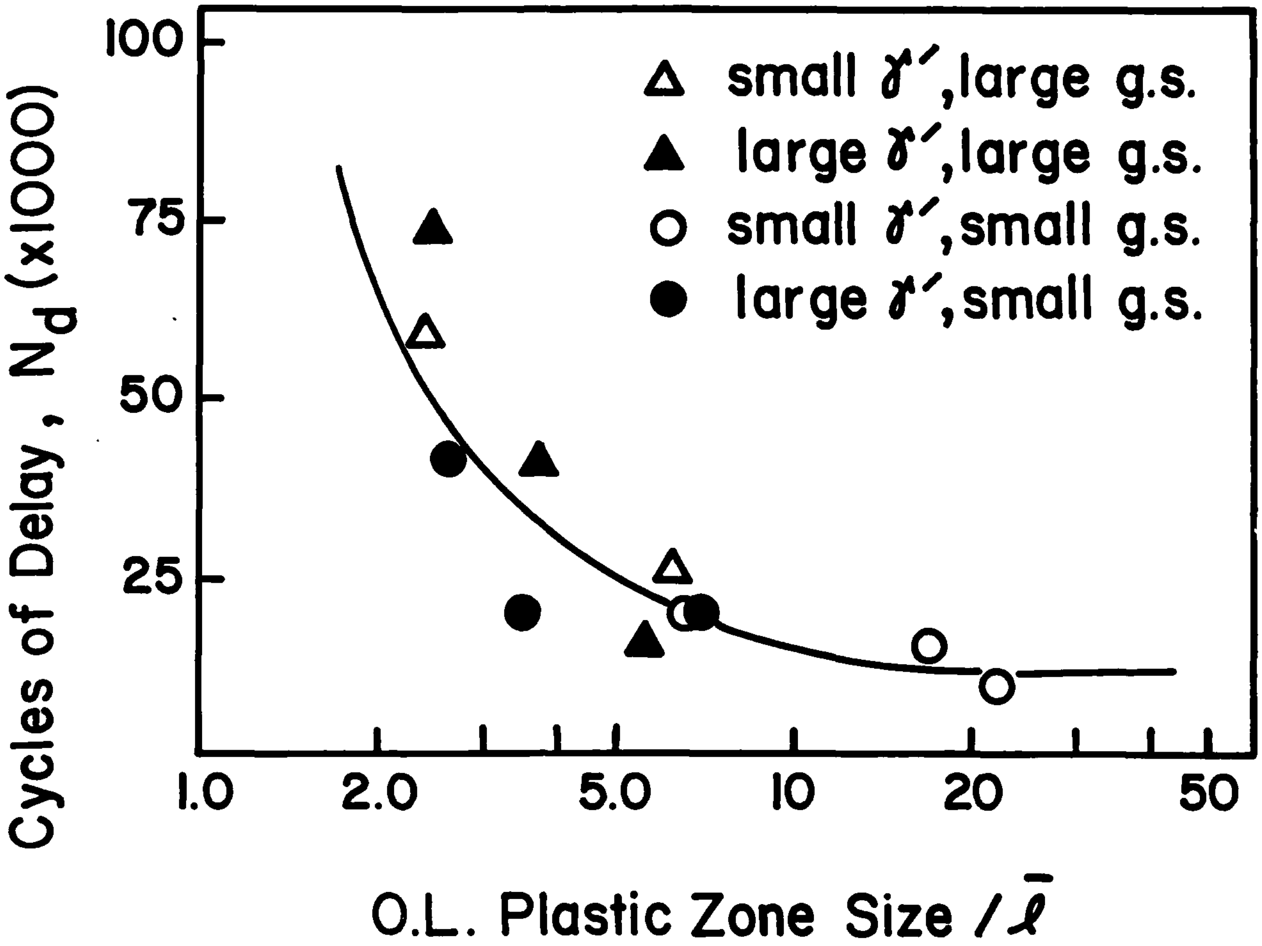

Fig. 1. Normalized data from Antolovich and Jayaraman[3] revealing influence of grain size on overload-induced cyclic delay.

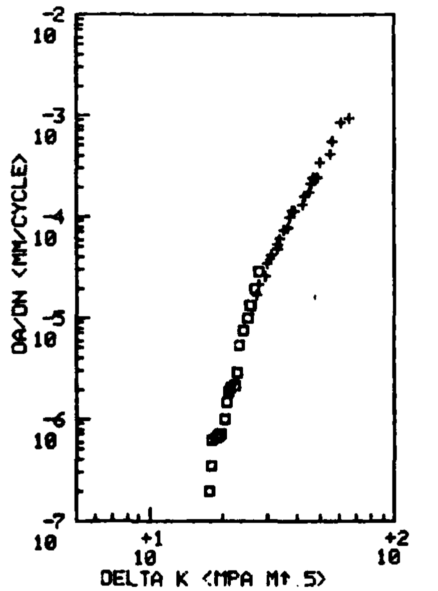

Fig. 2. FCP plot (R=0.1) of LC Astroloy HIP'd and solution treated at 1149°C and aged at 800°C/24 hrs.

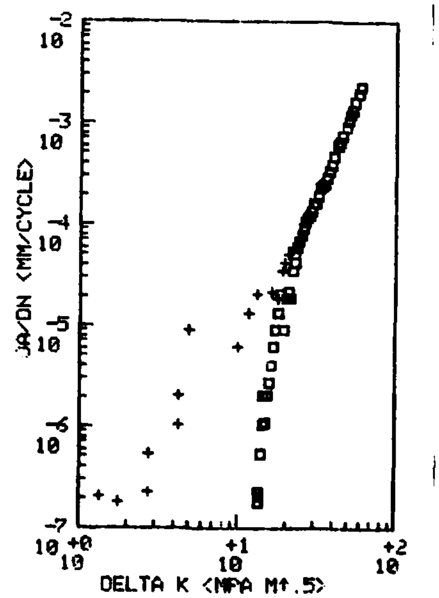

Fig. 3. FCP plot (R=0.5) of LC Astroloy HIP'd and solution treated at 1149°C and aged at 650°C/24 hrs.

APPENDIX A

Compliance Coefficients

Geometry: Round Compact Tension (Flat Face)

These results for the Round Compact Tension specimen (Figure 1) are based on the theoretical and experimental work of Newman[1] and Fisher and Buzzard[2]. A regression analysis written by D. Jablonski, was modified to run on an IBM PC microcomputer for the following compliance coefficient evaluation. The analysis used the following transfer function (U) for computing a/w as a function of EVB/P.

$$a/w = C_0 + C_1U_1 + C_2U_2^2 + C_3U_3^3 + C_4U_4^4 + C_5U_5^5$$

$$\text{where } U = \frac{1}{\left[\left(\frac{EVB}{P}\right)^{\frac{1}{2}} + 1.0\right]}$$

This transfer function was choosen because of the satisfactory results obtained by Saxena and Hudak[3] in their evaluation of the square compact and WOL specimens. Compliance for the Round Compact specimen is very similar to those just mentioned.

The values for the coefficients $C_0 + \ldots + C_5$ are given in Table I for the load line and front face (integral) positions, along with the standard error estimate.

Table I

Compliance Coefficients for Round Compact Tension Specimen
Analysis is valid for a/w values of 0.2 to 0.8

Position	C_0	C_1	C_2	C_3	C_4	C_5
Front Face (Integral)	1.02011	-5.92803	39.0251	-412.026	2086.90	-3880.88
Load Line	.975660	-3.00053	-11.7955	91.8882	-232.787	210.574

Standard Error Estimate

Front Face (Integral)	7.8×10^{-5}
Load Line	1.2×10^{-4}

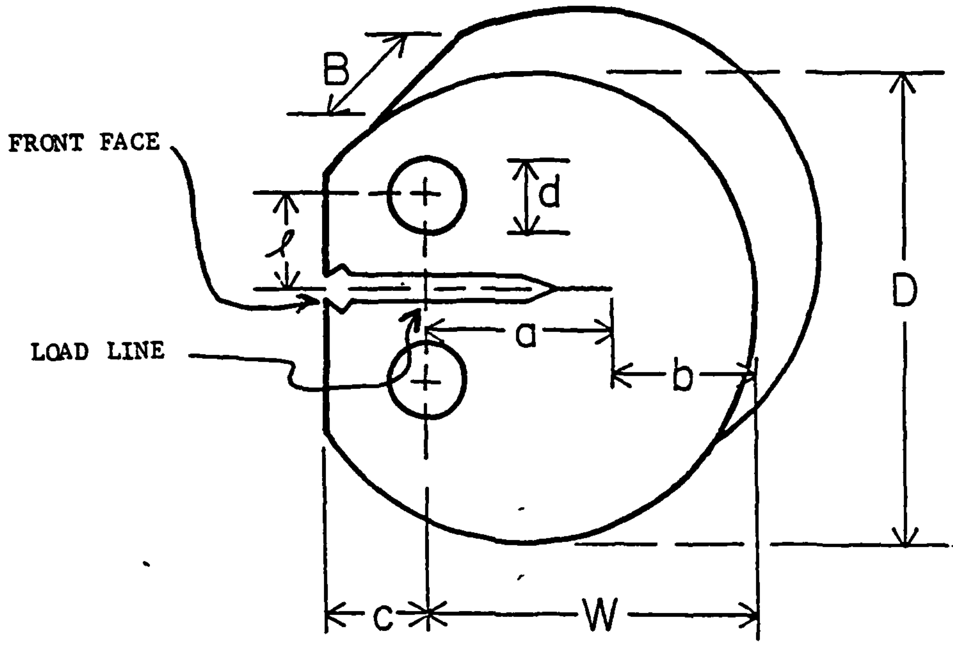

Fig. A1: Round compact tension specimen

References

(1) Newman, J. C., NASA TECM, MEM. 80174, October 1979, Langley Research Center.

(2) Fisher, D. M. and Buzzard, R. J., NASA TECH. MEM. 81379, November 1979, Lewis Research Center.

(3) Saxena, A. and Hudak, S. J., Int. J. Fracture, 14, No. 5, October 1978, p. 453.

Dissertations in Progress

Effect of Metallurgical and Load Spectrum Variables on Fatigue Crack Propagation in HIP'd Astroloy Nickel-Based Superalloy

Personnel

Richard W. Hertzberg, principal investigator
Jeffrey S. Crompton, research scientist
Robert S. Vecchio, research assistant

Coupling Activities

Crystal Newton, graduate fellow
(overload interaction effects in extruded aluminum alloys)